EXHIBIT D

ANALYTICAL METHOD FOR CHLORINATED BIPHENYL CONGENERS

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1.0 SCOPE AND APPLICATION

1.1 Method

The analytical method that follows is for the determination of Chlorinated Biphenyl (CB) congeners in water, soil, sediment, and tissue by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS). The method is based on EPA Method 1668A.

1.1.1 The CB congeners that can be determined by this method are the 12 Polychlorinated Biphenyls (PCBs) designated as toxic by the World Health Organization (WHO), plus the remaining 197 CB congeners, approximately 125 of which are resolved adequately on a SPB-octyl Gas Chromatographic (GC) column to be determined as individual congeners. The approximately 70 remaining congeners are determined as mixtures of isomers (coelutions).

1.1.2 The 12 PCBs designated as toxic by WHO and the earliest and latest eluted congeners at each Level Of Chlorination (LOC) are determined by the isotope dilution quantitation technique; the remaining congeners are determined by the Internal Standard quantitation technique.

1.1.3 This method allows determination of the PCB Toxicity Equivalent (TEQ_{PCB}) for the Toxics in a sample using Toxicity Equivalency Factors (TEFs).

1.1.4 This method also allows estimation of homologue totals by level of chlorination and estimation of total CB congeners in a sample by summation of the concentrations of the CB congeners and congener groups.

1.1.5 The list of 209 CB congeners is given in Table 1 with the Toxics and the LOC CB congeners identified.

1.2 Quantitation Levels

The detection limits and quantitation levels in this method are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The levels listed in Exhibit C are the Contract Required Quantitation Limits (CRQLs) that can be determined with no interferences present.

1.3 Qualitative Identification

The qualitative identification criteria (Section 11.1) include requirements for Retention Times (RTs), Relative Retention Times (RRTs), signal-to-noise (S/N) ratios, and limits on the ratios of the responses at two exact specified ions.

1.4 Qualification

The Contractor must demonstrate the ability to generate acceptable results using the procedure in Section 12.

2.0 SUMMARY OF METHOD

2.1 Extraction

2.1.1 Aqueous samples - Stable isotopically labeled analogs of the Toxics and labeled Level of Chlorination (LOC) Chlorinated Biphenyl (CB) Congeners are spiked into a 1 L sample. The sample is extracted using solid phase extraction (SPE), separatory funnel extraction (SFE), or continuous liquid/liquid extraction (CLLE).

2.1.2 Soil and Sediment samples - The labeled compounds are spiked into a sample containing 10 g (dry weight) of solids and extracted in a Soxhlet/Dean-Stark (SDS) Extractor. The extract is concentrated for cleanup.

2.1.3 Tissue samples (non-human) - A 20 g aliquot of a sample is homogenized, and a 10 g aliquot is spiked with the labeled compounds. The sample is mixed with anhydrous sodium sulfate, allowed to dry for 12-24 hours, and extracted for 18-24 hours using (1:1) (v/v) hexane/methylene chloride in a Soxhlet Extractor. The extract is evaporated to dryness, and the lipid content is determined.

2.2 Cleanup

After extraction, a Labeled Cleanup Standard is spiked into the extract which is then cleaned up using back-extraction with sulfuric acid and/or base, and gel permeation, silica gel, or Florisil chromatography. Activated carbon and High Performance Liquid Chromatography (HPLC) can be used for further isolation of specific congener groups. Before the cleanup procedures cited above, tissue extracts are cleaned up using an anthropogenic isolation column.

2.3 Analysis

After cleanup, the extract is concentrated to 20 µL. Immediately before injection, Labeled Internal Standards are added to each extract and an aliquot of the extract is injected into the Gas Chromatograph (GC). The analytes are separated by the GC and detected by a High Resolution ($10,000) Mass Spectrometer (HRMS). Two specified exact m/z ratios are monitored at each LOC throughout a predetermined Retention Time (RT) window.

An individual Toxic CB Congener is identified by comparing the GC RT and ion abundance ratio of two exact m/z ratios with the corresponding RT of an authentic standard and the theoretical or acquired ion abundance ratio of the two exact m/z ratios. Isomer specificity of the CB congeners is achieved using GC columns that resolve these congeners.

2.4 Quantitative Analysis

Quantitative analysis is performed in one of two ways using Selected Ion Current Profile (SICP) areas:

2.4.1 For the Toxics and the LOC CB Congeners, the GC/Mass Spectrometer (MS) is multi-point calibrated and the concentration is determined using the isotope dilution technique.

2.4.2 For all congeners other than the Toxics and LOC CB Congeners (if requested), the GC/MS is calibrated at a single concentration and the concentrations are determined using the Internal Standard technique.

2.4.3 For the labeled Toxics, labeled LOC CB Congeners, and the Cleanup Standards, the GC/MS is calibrated using replicates at a single concentration and the concentrations of these labeled compounds in samples are determined using the Internal Standard technique.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES AND CONTAMINATION

4.1 Sources of Contamination

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Environmentally abundant Chlorinated Biphenyl (CB) congeners, as well as toxic Congeners 105, 114, 118, 123, 156, 157, and 167 have been shown to be very difficult to completely eliminate from the laboratory at levels lower than the Estimated Method Detection Limits (EMDLs)(see Table 2). Baking of glassware in a kiln or furnace at 450-500°C may be necessary to remove these and other contaminants.

4.2 Glassware Cleaning

Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface.

4.2.1 Glassware should be rinsed with solvent and washed with a detergent solution after use. Sonication of glassware containing a detergent solution for approximately 30 sec. may aid in cleaning. Glassware with removable parts, particularly separatory funnels with fluoropolymer stopcocks, must be disassembled before detergent washing.

4.2.2 After detergent washing, glassware should be rinsed immediately, first with methanol, then with hot tap water. Another methanol rinse, then acetone, and then methylene chloride follow the tap water rinse.

4.2.3 Baking of glassware in a kiln or other high temperature furnace (300-500°C) may be warranted after particularly dirty samples are encountered. The kiln or furnace should be vented to prevent laboratory contamination by CB vapors. Baking should be minimized, as repeated baking of glassware may cause active sites on the glass surface that may irreversibly adsorb CB congeners.

4.2.4 Immediately before use, the Soxhlet apparatus should be pre-extracted with toluene for approximately 3 hours. The extraction apparatus should be rinsed with (80:20) (v/v) methylene chloride/toluene.

4.2.5 A separate set of glassware may be necessary to effectively preclude contamination when low-level samples are analyzed.

4.3 Reagents and Materials

All materials used in analysis must be demonstrated to be free from interferences by running reference matrix method blanks initially and with each sample batch (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples).

4.3.1 The reference matrix must simulate, as closely as possible, the sample matrix under a test. Ideally, the reference matrix should not contain the CB congeners in detectable amounts, but should contain potential interferents in the concentrations expected to be found in the samples to be analyzed.

4.3.2 When a reference matrix that simulates the sample matrix under a test is not available, reagent water (Section 7.6.1) can be used to simulate water samples; playground sand (Section 7.6.2) or white quartz sand (Section 7.3.2) can be used to simulate soils; and corn oil (Section 7.6.3) can be used to simulate tissues.

4.4 Interferences

Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations in several orders of magnitude higher than the CB congeners. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics, polychlorinated naphthalenes, and pesticides. Because this method measures very low levels of CB congeners, minimizing interferences is essential. The cleanup steps given in Section 10 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the CB congeners at the levels shown in Table 2.

4.5 Calibration Solutions

The EMDLs and Estimated Minimum Quantitation Levels (EMQLs) in Table 2 are the levels that can be achieved with normal laboratory backgrounds present. Many of the EMLs are greater than the equivalent concentrations of the calibration solutions. To prevent contamination of the calibration solutions with the backgrounds allowed by the EMLs, the calibration solutions must be prepared in an area free from CB contamination using glassware free from contamination. If these requirements cannot be met or are difficult to meet in the laboratory, the laboratory should prepare the calibration solutions in a contamination-free facility or have a vendor prepare the Calibration Standards and ensure a lack of contamination.

4.6 Lipids

The natural lipid content of tissue can interfere in the analysis of tissue samples for the CB congeners. The lipid contents of different species and portions of tissue can vary widely. Lipids are soluble to varying degrees in various organic solvents and may be present in sufficient quantity to overwhelm the column chromatographic cleanup procedures used for cleanup of sample extracts. Lipids must be removed by the anthropogenic isolation column procedure in Section 10.5.5, followed by the Gel Permeation Chromatography (GPC) procedure in Section 10.5.1 Florisil cleanup (Section 10.5.6) is recommended as an additional step.

5.0 SAFETY

5.1 Toxicity

The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined; however, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.1.1 Chlorinated Biphenyl (CB) congeners have been tentatively classified as known or suspected human or mammalian carcinogens. Based on the available toxicological and physical properties of the CB congeners, only highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks should handle pure standards.

5.1.2 It is recommended that the laboratory purchase dilute standard solutions of the analytes in this method. However, if primary solutions are prepared, they must be prepared in a hood, and a NIOSH/MESA-approved toxic gas respirator must be worn when high concentrations are handled.

5.2 Occupational Safety and Health Administration (OSHA) Requirements

The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDSs) should also be made available to all personnel involved in these analyses. It is also suggested that the laboratory perform personal hygiene monitoring of each analyst who uses this method and that the results of this monitoring be made available to the analyst.

5.3 Sample Handling

The pure CB congeners and samples suspected to contain these compounds are handled using essentially the same techniques employed in handling radioactive or infectious materials. Well-ventilated and controlled access laboratories are required. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or Labor, many of which have an industrial health service. Each laboratory must develop a strict safety program for handling these compounds.

5.3.1 Facility - When the divided samples (dusts, soils, dry chemicals) are handled, all operations (including removal of samples from sample containers, weighing, transferring, and mixing) should be performed in a glove box demonstrated to be leak-tight or in a fume hood demonstrated to have adequate air flow. Gross losses to the laboratory ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in the case of an accident.

5.3.2 Protective Equipment - Disposable plastic gloves, apron or laboratory coat, safety glasses or mask, and a glove box or fume hood adequate for radioactive work should be used. During analytical operations that may give rise to aerosols or dusts, personnel should wear respirators equipped with activated carbon filters. Eye protection (preferably full-face shields) must be worn while working with exposed samples or pure analytical standards. Latex gloves are commonly used to reduce exposure of the hands. When handling samples suspected or known to contain high concentrations of the CB congeners, an additional set of gloves can also be worn beneath the latex gloves.

5.3.3 Training - Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.3.4 Personal Hygiene - Hands and forearms should be washed thoroughly after each manipulation and before breaks (coffee, lunch, and shift).

5.3.5 Confinement - Isolated work areas posted with signs, segregated glassware and tools, and plastic absorbent paper on bench tops will aid in confining contamination.

5.3.6 Effluent Vapors - The effluent of the sample splitter from the Gas Chromatograph (GC) and from roughing pumps on the Mass Spectrometer (MS) should pass through either a column of activated charcoal or be bubbling through a trap containing oil or high-boiling alcohol to condense CB vapors.

5.3.7 Waste Handling - Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors and other personnel should be trained in the safe handling of waste.

5.4 Decontamination

5.4.1 Decontamination of Personnel - Use any mild soap with plenty of scrubbing action.

5.4.2 Glassware, Tools, and Surfaces - Chlorothene NU Solvent is a less toxic solvent that should be effective in removing CB congeners. Satisfactory cleaning may be accomplished by rinsing with Chlorothene, then washing with any detergent and water. If glassware is first rinsed with solvent, the wash water may be disposed of in the sewer. Given the cost of disposal, it is prudent to minimize solvent wastes.

5.4.3 Laundry - Clothing known to be contaminated should be collected in plastic bags. Persons that convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows of the potential problem. The washer should be run through a cycle before being used again for other clothing.

5.4.4 Wipe Tests - A useful method of determining cleanliness of work surfaces and tools is to perform a wipe test of the surface suspected of being contaminated.

5.4.4.1 Using a piece of filter paper moistened with Chlorothene or other solvent, wipe an area approximately 10 x 10 cm.

5.4.4.2 Extract and analyze the wipe by GC with an Electron Capture Detector (ECD) or by this method.

5.4.4.3 Using the area wiped (e.g., 10 x 10 cm = 0.01 m^2), calculate the concentration in µg/m^2. A concentration less than 1 µg/m^2 indicates acceptable cleanliness; anything higher warrants further cleaning. Concentrations more than 100 µg/m^2 constitute an acute hazard and requires prompt cleaning before further use of the equipment or workspace, and indicate that unacceptable work practices have been employed.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustration purposes only and no endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here. Meeting the performance requirements of this method is the responsibility of the laboratory.

6.1 Glassware Cleaning

6.1.1 Laboratory sink with an overhead fume hood.

6.1.2 Kiln - Capable of reaching 450°C within 2 hours and maintaining 450-500°C within ±10°C, with a temperature controller and safety switch.

6.2 Sample Preparation

6.2.1 A laboratory fume hood of sufficient size to contain the sample preparation equipment listed below.

6.2.2 Glove Box (optional).

6.2.3 Tissue Homogenizer - With stainless steel shaft and blade.

6.2.4 Meat Grinder - With 3 to 5 mm holes in inner plate.

6.2.5 Equipment for Determining Percent Moisture

6.2.5.1 Oven - Capable of maintaining a temperature of 110 ±5°C.

6.2.5.2 Desiccator

6.2.6 Balances

6.2.6.1 Analytical - Capable of weighing 0.1 mg.

6.2.6.2 Top loading - Capable of weighing 10 mg.

6.3 Extraction Apparatus

6.3.1 Aqueous Samples

6.3.1.1 pH meters, with a combination glass electrode.

6.3.1.2 pH paper, wide range.

6.3.1.3 Graduated Cylinder, 1 L capacity.

6.3.1.4 Liquid/Liquid Extraction - Separatory funnels, 250, 500, and 2000 mL, with fluoropolymer stopcocks.

6.3.1.5 Solid Phase Extraction (SPE)

6.3.1.5.1 1 L filtration apparatus, including glass funnel, frit support, clamp, adapter, stopper, filtration flask, and vacuum tubing. For wastewater samples, the apparatus should accept 90 or 144 mm disks. For drinking water or other samples containing low solids, smaller disks may be used.

6.3.1.5.2 Vacuum Source - Capable of maintaining 25 in. Hg, equipped with shutoff valve and vacuum gauge.

6.3.1.5.3 Glass-Fiber Filter - Whatman GMF 150 (or equivalent), 1 micron pore size, to fit filtration apparatus in Section 6.3.1.5.1.

6.3.1.5.4 SPE disk containing octadecyl (C_{18}) bonded silica uniformly enmeshed in an inert matrix, to fit filtration apparatus in Section 6.3.1.5.1.

6.3.1.6 Continuous liquid/liquid extraction (CLLE) - Fluoropolymer or glass connecting joints and stopcocks without lubrication, 1.5 to 2 L capacity.

6.3.2 Soil/Sediment Samples

Soxhlet/Dean-Stark (SDS) Extractor for solid samples

6.3.2.1 Soxhlet - 50 mm ID, 200 mL capacity with 500 mL round bottom flask.

6.3.2.2 Thimble - 43 × 123 to fit Soxhlet.

6.3.2.3 Moisture Trap - Dean-Stark or Barret with fluoropolymer stopcock, to fit Soxhlet Extractor.

6.3.2.4 Heating Mantle - Hemispherical, to fit 500 mL round-bottom flask.

6.3.2.5 Variable Transformer - 110-volt, 10 amp.

6.3.3 Tissue Samples

6.3.3.1 Beakers - 400 to 500 mL.

6.3.3.2 Spatulas - Stainless steel.

6.4 Filtration Apparatus

6.4.1 Borosilicate Glass Wool - Solvent-extracted using a Soxhlet or SDS Extractor for 3 hours minimum.

6.4.2 Glass Funnel - 125 to 250 mL.

6.4.3 Glass-Fiber Filter Paper - Whatman GF/D (or equivalent), to fit glass funnel in Section 6.4.2.

6.4.4 Drying Column - 15 to 20 mm ID borosilicate chromatographic column equipped with a coarse-glass frit or glass wool plug.

6.4.5 Buchner Funnel - 15 cm.

6.4.6 Glass-Fiber Filter Paper for the Buchner Funnel, as listed above.

6.4.7 Filtration Flasks - 1.5 to 2.0 L, with a side arm.

6.4.8 Pressure Filtration Apparatus.

6.5 Centrifuge Apparatus

6.5.1 Centrifuge - Capable of rotating 500 mL centrifuge bottles or 15 mL centrifuge tubes at 5,000 rpm minimum.

6.5.2 Centrifuge Bottles - 500 mL, with screw-caps, to fit centrifuge.

6.5.3 Centrifuge Tubes - 12 to 15 mL, with screw-caps, to fit centrifuge.

6.6 Cleanup Apparatus

6.6.1 Automated Gel Permeation Chromatograph (GPC)

6.6.1.1 Column - 600-700 mm long × 25 mm ID glass, packed with 70 g of 200-400 mesh SX-3 Bio-beads (Bio-Rad Laboratories, Richmond, CA, or equivalent).

6.6.1.2 Syringe - 10 mL, with Luer-Lok fitting.

6.6.1.3 Syringe Filter Holder - Stainless steel with glass-fiber or fluoropolymer filters.

6.6.1.4 UV Detectors - 254 nm, preparative or semi-preparative flow cell.

6.6.2 Reverse-Phase High-Performance Liquid Chromatograph

6.6.2.1 Pump.

6.6.2.2 Injector.

6.6.2.3 Port Switching Valve.

6.6.2.4 Column - Hypercarb, 100 x 4.6 mm, 5 µm particle size, Keystone Scientific, or equivalent.

6.6.2.5 Detector - Operated at 0.02 AUFS at 235 nm.

6.6.2.6 Fraction Collector.

6.6.3 Pipets

6.6.3.1 Disposable, Pasteur, 150 mm long × 5 mm ID.

6.6.3.2 Disposable, serological, 50 mL (8 to 10 mm ID).

6.6.4 Glass Chromatographic Columns

6.6.4.1 150 mm long × 8 mm ID, with coarse-glass frit or glass wool plug and 250 mL reservoir.

6.6.4.2 200 mm long × 15 mm ID, with coarse-glass frit or glass wool plug and 250 mL reservoir.

6.6.4.3 300 mm long x 22 mm ID, with coarse-glass frit, 300 mL reservoir, and glass or fluoropolymer stopcock.

6.6.5 Oven - For baking and storage of adsorbents, capable of maintaining a constant temperature (±5°C) in the range of 105-250°C.

6.7 Concentration Apparatus

6.7.1 Rotary Evaporator - Equipped with a variable temperature water bath.

6.7.1.1 Vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge.

6.7.1.2 A recirculating water pump and chiller are recommended, as use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary.

6.7.1.3 Round-bottom Flask - 100 mL and 500 mL or larger, with ground-glass fitting compatible with the rotary evaporator.

6.7.2 Kuderna-Danish (K-D) Concentrator

6.7.2.1 Concentrator Tube - 10 mL, graduated with calibrations verified. Ground-glass stopper (Size 19/22 joint) is used to prevent evaporation of extracts.

6.7.2.2 Evaporation Flask - 500 mL attached to concentrator tube with springs.

6.7.2.3 Snyder Column - Three-ball macro.

6.7.2.4 Boiling Chips

6.7.2.4.1 Glass or Silicon Carbide - Approximately 10/40 mesh, extracted with methylene chloride and baked at 450°C for 1 hour minimum.

6.7.2.4.2 Fluoropolymer (optional) - Extracted with methylene chloride.

6.7.2.5 Water Bath - Heated, with concentric ring cover, capable of maintaining a temperature within ±2°C, installed in a fume hood.

6.7.3 Nitrogen Blowdown Apparatus - Equipped with water bath controlled in the range of 30-60°C, installed in a fume hood.

6.7.4 Sample Vials

6.7.4.1 Amber glass, 2 to 5 mL with fluoropolymer-lined screw-cap.

6.7.4.2 Glass, 0.3 mL, conical, with fluoropolymer-lined screw or crimp cap.

6.8 Gas Chromatograph

Must have splitless or on-column injection port for a capillary column, a temperature program with isothermal hold, and must meet all of the performance specifications in Section 9.

6.8.1 GC Column - Any GC column or column system (two or more columns) that provide unique resolution and identification of the Toxics for determination of a PCB Toxicity Equivalent (TEQ_{PCB}) using Toxicity Equivalency Factors (TEFs). Isomers may be unresolved so long as they have the same TEF and Response Factor (RF) and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-octyl column (Section 6.8.1.2) achieves unique GC resolution of all Toxics except congeners with IUPAC Numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and RF.

6.8.1.1 If a SPB-octyl column is used, it must meet the specification in Section 6.8.1 and the following additional specifications:

6.8.1.1.1 The column must uniquely resolve Congeners 34 from 23 and 187 from 182, and Congeners 156 and 157 must coelute within 2 sec. at the peak maximum. Unique resolution means a valley height less than 40% of the shorter of the two peaks that result when the diluted combined 209-Congener Standard Solution (Section 7.10.2.2) is analyzed.

6.8.1.1.2 The column must be replaced when any of the criteria in Sections 6.8.1 - 6.8.1.1.1 are not met.

6.8.1.2 Suggested Column - 30(±5) m long × 0.25 (±0.02) mm ID; 0.25 µm film SPB-octyl. This column is capable of meeting the requirements in Sections 6.8.1 - 6.8.1.1.1.

NOTE: The SPB-octyl column is subject to rapid degradation when exposed to oxygen. The analyst should exclude oxygen from the carrier gas, eliminate air leaks, and cool the injector, column, and transfer line before opening the column to the atmosphere. For further information on precluding oxidation, contact the column manufacturer.

6.9 Mass Spectrometer - 28 to 40 eV electron impact ionization, must be capable of selectively monitoring a minimum of 22 exact m/z ratios minimum at high resolution ($10,000) during a period less than 1.5 sec. Must meet all of the performance specifications in Section 9.

6.10 GC/MS Interface - The Mass Spectrometer (MS) must be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.

6.11 Data System — Capable of collecting, recording, storing, and processing MS data.

6.11.1 Data Acquisition - The signal at each exact m/z must be collected repetitively throughout the monitoring period and stored on a mass storage device.

6.11.2 RFs and Multi-point Calibrations - The data system must record and maintain lists of RFs (response ratios for isotope dilution) and multi-point calibrations. Computations of Relative Standard Deviation (RSD) are to be used to test calibration linearity.

7.0 REAGENTS AND STANDARDS

7.1 pH Adjustment and Back-Extraction

7.1.1 Potassium Hydroxide - Dissolve 20 g reagent grade KOH in 100 mL reagent water.

7.1.2 Sulfuric acid - Reagent Grade (specific gravity 1.84)

7.1.3 Hydrochloric acid, 6N (1:1) (v/v) - Add 500 mL concentrated HCl to 400 mL reagent water and dilute to 1 L.

7.1.4 Sodium Chloride - Dissolve 5 g reagent grade NaCl in 100 mL reagent water.

7.2 Solution Drying and Evaporation

7.2.1 Solution Drying - Sodium sulfate, reagent grade, granular, anhydrous, rinsed with methylene chloride (20 mL/g), baked at 400°C for 1 hour minimum, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw-cap that prevents moisture from entering. If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of the reagent is not suitable for use and should be discarded. Extraction with methylene chloride (as opposed to simple rinsing) and baking at a lower temperature may produce sodium sulfate that is suitable for use.

7.2.2 Tissue Drying - Sodium sulfate, reagent grade, powdered, treated and stored as in Section 7.2.1.

7.2.3 Pre-purified Nitrogen

7.3 Extraction

7.3.1 Solvents - Acetone, toluene, cyclohexane, hexane, methanol, methylene chloride, iso-octane, and nonane; distilled in glass, pesticide quality, lot-certified to be free of interferences.

NOTE: Some solvents (e.g., iso-octane and nonane) may need to be redistilled to eliminate Chlorinated Biphenyl (CB) backgrounds.

7.3.2 White quartz sand, 60/70 mesh - For Soxhlet/Dean-Stark (SDS) extraction. Bake at 450°C for a minimum of 4 hours.

7.4 GPC Calibration Solution - Prepare a solution containing 2.5 mg/mL corn oil, 0.05 mg/mL bis(2-ethylhexyl) phthalate (BEHP), 0.01 mg/mL methoxychlor, 0.002 mg/mL perylene, and 0.008 mg/mL sulfur, or at concentrations appropriate to the response of the detector.

7.5 Absorbents for a Sample Cleanup

7.5.1 Silica Gel

7.5.1.1 Activated Silica Gel - 100-200 mesh, rinsed with methylene chloride, baked at 180°C for a minimum of 1 hour, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw-cap that prevents moisture from entering.

7.5.1.2 Acidic Silica Gel (30% w/w) - Thoroughly mix 44 g of concentrated sulfuric acid with 100 g of activated silica gel in a clean container. Break up aggregates with a stirring rod until a uniform mixture is obtained. Store in a screw-capped bottle with a fluoropolymer-lined cap.

7.5.1.3 Basic Silica Gel - Thoroughly mix 30 g of a 1 N sodium hydroxide with 100 g of activated silica gel in a clean container. Break up aggregates with a stirring rod until a uniform mixture is obtained. Store in a screw-capped bottle with a fluoropolymer-lined cap.

7.5.1.4 Potassium Silicate

7.5.1.4.1 Dissolve 56 g of a high purity potassium hydroxide in 300 mL of methanol in a 750 to a 1000 mL flat-bottom flask.

7.5.1.4.2 Add 100 g of activated silica gel (Section 7.5.1.1) and a stirring bar, then stir on an explosion-proof hot plate at 60-70°C for 1-2 hours.

7.5.1.4.3 Decant the liquid and rinse the potassium silicate twice with 100 mL portions of methanol, followed by a single rinse with 100 mL of methylene chloride.

7.5.1.4.4 Spread the potassium silicate on solvent-rinsed aluminum foil and dry for 2-4 hours in a hood.

7.5.1.4.5 Activate overnight at 200-250°C before use.

7.5.2 Carbon

7.5.2.1 Carbopak C - (Supelco 1-0258, or equivalent)

7.5.2.2 Celite 545 - (Supelco 2-0199, or equivalent)

7.5.2.3 Thoroughly mix 18 g Carbopak C and 18 g Celite 545 to produce a 50% w/w mixture. Activate the mixture at 130°C for a minimum of 6 hours. Store in a desiccator.

NOTE: The carbon column has been included in this method to allow separation of coplanar Congeners 77, 126, and 169 from other congeners and interferences, should such separation be desired.

7.5.3 Anthropogenic Isolation Column - Pack the column in Section 6.6.4.3 from bottom to top with the following:

7.5.3.1 2 g activated silica gel (Section 7.5.1.1)

7.5.3.2 2 g activated potassium silicate (Section 7.5.1.4)

7.5.3.3 2 g granular anhydrous sodium sulfate (Section 7.2.1)

7.5.3.4 10 g acidic silica gel (Section 7.5.1.2)

7.5.3.5 2 g granular anhydrous sodium sulfate

7.5.4 Florisil Column

7.5.4.1 Florisil - PR grade, 60-100 mesh. Alternatively, prepackaged Florisil columns may be used. Use the following procedure for Florisil activation and column packing.

7.5.4.1.1 Fill a clean 1-2 L bottle 1/2 to 2/3 full with Florisil and place in an oven at 130-150°C for a minimum of 3 days to activate the Florisil.

7.5.4.1.2 Immediately before use, dry pack a 300 mm x 22 mm ID glass column (Section 6.6.4.3) bottom to top with 0.5 - 1.0 cm of warm to hot anhydrous sodium sulfate (Section 7.2.1), 10 - 10.5 cm of warm to hot activated Florisil (Section 7.5.4.1.1), and 1-2 cm of warm to hot anhydrous sodium sulfate. Allow the column to cool and wet immediately with 100 mL of n-hexane to prevent moisture from entering.

7.5.4.2 Using the procedure in Section 10.5.6.3, establish the elution pattern for each carton of Florisil or each lot of Florisil columns received.

7.6 Reference Matrices

Matrices that the CB congeners and interfering compounds are not detected in by this method.

7.6.1 Reagent Water - Water demonstrated to be free from the analytes of interest and potentially interfering substances.

7.6.2 Soil Reference Matrix - Playground sand or similar material. Prepared by extraction with methylene chloride and/or baking at 450°C for a minimum of 4 hours.

7.6.3 Tissue Reference Matrix - Corn or other vegetable oil.

7.7 Standard Solutions

Prepare standard solutions from materials of known purity and composition or purchase as solutions or mixtures with certification to their purity, concentration, and authenticity. If the chemical purity is 98% or greater, the weight may be used without correction to calculate the concentration of the standard. Observe the safety precautions in Section 5 and the recommendation in Section 5.1.2.

7.7.1 For preparation of stock solutions from neat materials, dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 1 to 2 mg of PCB 126 to three significant figures in a 10 mL ground-glass-stoppered volumetric flask and fill to the mark with nonane. After the compound is completely dissolved, transfer the solution to a clean 15 mL vial with a polytetrafluoroethylene (PTFE)-lined cap.

7.7.2 When not being used, store standard solutions in the dark at room temperature in screw-capped vials with PTFE-lined caps. Place a mark on the vial at the level of the solution so that solvent loss by evaporation can be detected. Replace the solution if solvent loss has occurred.

7.8 Native (Unlabeled) Stock Solutions

7.8.1 Native Toxics/Level of Chlorination (LOC) Stock Solution — Prepare to contain the native Toxics and LOC CB Congeners at the concentrations shown in Table 3, or purchase.

7.8.2 Native 209 CB Congener Stock Solutions — Solutions containing CB congeners to calibrate the High Resolution Gas Chromatograph/Mass

Spectrometer (HRGC/HRMS). Prepare solutions that will allow separation of all 209 congeners on the selected column, or purchase.

7.8.3 Stock solutions should be checked for signs of degradation before the preparation of Calibration or Performance Test Standards. Reference Standards that can be used to determine the accuracy of standard solutions are available from several vendors.

7.9 Labeled Compound Stock Solutions (Table 3)

7.9.1 Labeled Toxics/LOC/Window-Defining Congeners Stock Solution — Prepare in iso-octane or nonane at the concentrations in Table 3, or purchase.

7.9.2 Labeled Cleanup Standard Stock Solution - Prepare labeled CB Congeners 28, 111, and 178 in iso-octane or nonane at the concentration shown in Table 3, or purchase.

7.9.3 Labeled Injection Internal Standard Stock Solution — Prepare labeled CB Congers 9, 52, 101, 138, and 194 in nonane or iso-octane at the concentrations shown in Table 3, or purchase.

7.10 Calibration Standards (CSs)

7.10.1 Calibration Standards — Combine and dilute the solutions in Sections 7.8.1 and 7.9 to produce the Calibration Standards in Table 5. These standards may also be purchased from commercial sources. If a 6-point calibration is used, prepare the CS0.2 Standard, or purchase. These solutions permit the Relative Response (RR) (labeled to native) and a Relative Response Factor (RRF) to be measured as a function of concentration. The CS3 Standard is used for calibration verification.

7.10.2 Solutions of Congener Mixes

7.10.2.1 Diluted Individual Solutions

7.10.2.1.1 The individual solutions, when analyzed individually, allow resolution of all 209 congeners on the SPB-octyl column, and are used for establishing Retention Time (RT) and other data for each congener.

7.10.2.1.2 Individually combine an aliquot of each individual mix stock solution (Section 7.8.2) with an aliquot of the Labeled Toxics/LOC/Window-Defining Congeners Stock Solution (Section 7.9.1), the Labeled Cleanup Standard Stock Solution (Section 7.9.2), and the Labeled Injection Internal Standard Stock Solution (Section 7.9.3) to produce concentrations of 100 ng/mL for the labeled compounds and 25, 50, and 75 ng/mL for the MoCB - TrCB, TeCB -HpCB, and OcCB - DeCB Congeners, respectively, as shown in Table 3.

7.10.2.2 Diluted Combined 209-Congener Standard Solution

7.10.2.2.1 This solution combines the individual mixes with the labeled compounds to allow a single-point calibration of the congeners not included in the multi-point calibration, and establishes an average Response Factor (RF) for the coeluting Isomeric Congeners.

7.10.2.2.2 Combine aliquots of the individual mixes with an aliquot of the Labeled Toxics/LOC/Window-Defining Congeners Stock Solution

(Section 7.9.1), the Labeled Cleanup Standard Stock Solution (Section 7.9.2), and the Labeled Injection Internal Standard Stock Solution (Section 7.9.3) to produce the same concentrations as in the diluted individual mix solutions (Section 7.10.2.1.2 and Table 3).

7.11 Native Toxics/LOC Standard Spiking Solution

Used for determining Initial Precision and Recovery (IPR) Standards (Section 12.3). Dilute the Native Toxics/LOC stock solution (Section 7.8.1) with acetone to produce a concentration of the Toxics at 1 ng/mL, as shown in Table 3. When 1 mL of this solution spiked into the IPR (Section 9.2.1) and concentrated to a final volume of 20 µL, the concentration in the final volume will be 50 ng/mL (50 pg/µL).

7.12 Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution

This solution is spiked into each sample (Section 10.1), blanks (Section 12.2), and the IPR (Section 12.3) to measure recovery. Dilute the Labeled Toxics/LOC/Window-Defining Congeners Stock Solution (Section 7.9.1) with acetone to produce a concentration of the labeled compounds at 2 ng/mL, as shown in Table 3. When 1 mL of this solution is spiked into an IPR, blank, or sample and concentrated to a final extract volume of 20 µL, the concentration in the final extract volume will be 100 ng/mL (100 pg/µL). Prepare only the amount necessary for each reference matrix with each sample batch.

7.13 Labeled Cleanup Standard Spiking Solution

This solution is spiked into each extract before cleanup to measure the efficiency of the cleanup process. Dilute the Labeled Cleanup Standard Stock Solution (Section 7.9.2) in methylene chloride to produce a concentration of the Cleanup Standards at 2 ng/mL, as shown in Table 3. When 1 mL of this solution is spiked into a sample extract and concentrated to a final volume of 20 µL, the concentration in the final volume will be 100 ng/mL (100 pg/µL).

7.14 Labeled Internal Standard Spiking Solution

This solution is added to each concentrated extract before injection into the HRGC/HRMS. Dilute the Labeled Internal Standard Stock Solution (Section 7.9.3) in nonane to produce a concentration of the Injection Internal Standards at 1000 ng/mL, as shown in Table 3. When 2 µL of this solution is spiked into the 20 µL extract, the concentration of each Internal Standard will be nominally 100 ng/mL (100 pg/µL).

NOTE: The addition of 2 µL of the Labeled Internal Standard Spiking Solution to a 20 µL final extract has the effect of diluting the concentration of the components in the extract by 10%. Provided all calibration solutions and all extracts undergo this dilution as a result of adding the Labeled Injection Internal Standard Spiking Solution, the effect of the 10% solution is compensated, and correction for this dilution should not be made.

7.15 Retention Time (RT) Window-Defining Mixture (WDM)

Used to define the beginning and ending RTs for congeners at each LOC. The mixture must contain an appropriate amount of Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution [CS1 or CS3 may be used as the WDM (Section 9.5)].

7.16 Stability of Solutions

Standard solutions used for quantitative purposes (Sections 7.9 - 7.14) should be assayed periodically (e.g., every 6 months) against Standard Reference Materials (SRMs) from the NIST (if available), or certified reference materials from a source that will attest to the authenticity and concentration, to assure that the composition and concentrations have not changed.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Aqueous grab and composite samples must be collected in amber glass containers following conventional sampling practices. If residual chlorine is present, 80 mg sodium thiosulfate per liter of water should have been added and the pH should be adjusted to 2-3 with sulfuric acid. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine. All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until sample receipt at the laboratory.

8.1.2 Soil samples are collected as grab samples in amber glass jars. All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until receipt at the laboratory. Refer to Section 10.1.2 for oily and multi-phase samples.

8.1.3 Tissue samples collected in the field should be wrapped in aluminum foil, and must be maintained at a temperature less than 4°C from the time of collection until receipt at the laboratory. Ideally, tissues should be frozen upon collection and shipped to the laboratory under dry ice.

8.2 Procedures for Sample Storage

8.2.1 Maintain aqueous samples in the dark at 4°C (±2°C) from time of receipt until extraction.

8.2.2 Store soil samples in the dark at less than -10°C.

8.2.3 Tissue samples must be frozen upon receipt at the laboratory and stored in the dark at less than -10°C until prepared. Unused sample portions and unused homogenized tissues must be stored in the dark at less than 10°C.

8.2.4 Samples, sample extracts, and standards must be stored separately in the dark.

8.3 Contract Required Holding Times

The technical holding time for Chlorinated Biphenyl (CB) congeners, water or soil samples, stored in the dark at 4°C (±2°C) is one year. The technical holding time for CB congener tissue samples stored in the dark at less than -10°C is one year.

9.0 CALIBRATION AND STANDARDIZATION

9.1 High Resolution Gas Chromatograph (HRGC)

Establish the operating conditions necessary to meet the Retention Times (RTs) and Relative Retention Times (RRTs) for the Chlorinated Biphenyl Congeners in Table 2.

9.1.1 Suggested Gas Chromatograph (GC) Operating Conditions:

Injector temperature:	270°C
Interface temperature:	290°C
Initial temperature:	75°C
Initial time:	2 min.
Temperature program:	75-150°C at 15°C/min.
	150-290°C at 2.5°C/min.
Final time:	1 min.

NOTE: All portions of the column that connects the GC to the ion source should remain at or above the interface temperature (specified above) during analysis to preclude condensation of less volatile compounds.

The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, Initial Precision and Recovery (IPR) Standards, and samples.

9.1.2 Retention Time Calibrations for the CB Congeners

9.1.2.1 Separately inject each of the diluted individual congener solutions (Section 7.10.2.1.2). Establish the beginning and ending RTs for the scan descriptors in Table 7. Scan descriptors other than those listed in Table 7 may be used, provided that the Contract Required Quantitation Limits (CRQLs) in Exhibit C are met. Store the RT and RRT for each congener in the data system.

9.1.2.2 Inject the diluted combined 209-Congener Standard Solutions (Section 7.10.2.2 and Table 5). Adjust the chromatographic conditions and scan descriptors until the RT and RRT for all congeners are within the windows in Table 2 and the column performance specifications in Sections 6.8 are met. If an alternate column is used, adjust the conditions for that column. If column performance is unacceptable, optimize the analysis conditions or replace the column and repeat the performance tests. Confirm that the scan descriptor changes at times when CB congeners do not elute.

9.1.2.3 After the column performance tests are passed (Section 9.1.2.1 - 9.1.2.2), store the RT and RRT for the resolved congeners and the RT and RRT for the isomeric Congeners that coelute.

9.2 High Resolution Mass Spectrometer (HRMS)

9.2.1 Using perfluorokerosene (PFK) and a molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley) at m/z 330.9792 or any other significant PFK fragment in the range of 300-350. For each descriptor (Table 7), monitor and record the resolution and exact m/z ratios of 3 to 5 reference peaks covering the mass range of the descriptor. The level of PFK metered into the HRMS during analyses should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Different lots and types of PFK can contain varying levels of contamination, and excessive PFK may cause noise problems and contamination of the ion source necessitating increased frequency of source cleaning.

9.2.2 The analysis time for CB congeners may exceed the long-term mass stability of the Mass Spectrometer (MS). Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, mass-drift correction is mandatory and a lock-mass m/z from PFK is used for drift correction. The lock-mass m/z is dependent on the exact m/z ratios monitored within each descriptor, as shown in Table 7. The deviation between the exact m/z and the theoretical m/z (Table 7) for each exact m/z monitored must be less than 5 ppm.

9.2.3 Obtain a Selected Ion Current Profile (SICP) at the two exact m/z ratios specified in Table 7 and at $10,000 resolving power at each Level of Chlorination (LOC) or the native congeners and congener groups and for the labeled congeners. Due to the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolutions throughout the mass range during the function. Therefore, resolution must be $8,000 throughout the mass range and must be $10,000 in the center of the mass range for each function.

9.2.4 If the HRMS has the capability to monitor resolution during the analysis, it is acceptable to terminate the analysis when the resolution falls below the minimum to save reanalysis time.

9.3 Summary of HRGC/HRMS System Performance Check

9.3.1 The HRMS system must be tuned to meet the minimum static resolving power using PFK, and the resolution of the HRGC system must be verified by the analysis of the descriptor switching times set using the appropriate WDM (CS1 or CS3 Standard).

9.3.2 At the beginning of each 12-hour shift and before analysis of any samples, blanks, or Calibration Standards, the Contractor must establish that the HRGC/HRMS system meets the static resolving power for PFK, and that the beginning and ending RTs for congeners at each LOC is defined using the WDM.

9.3.3 The LOC/Window-Defining Congeners are also used to set the descriptor switching times such that congeners that elute from the HRGC during a given RT window will also be those congeners for which the ions are monitored.

9.4 HRMS System Tune

9.4.1 Frequency of HRMS System Tune

9.4.1.1 The PFK tune must be performed prior to the analysis of Calibration Standards, Initial Precision and Recovery (IPR) Standards, samples, and blanks within each 12-hour period.

9.4.1.2 The 12-hour period for the HRGC/HRMS system performance check does not begin until the HRMS system is tuned to meet the minimum required resolving power of 10,000 (10% valley) at m/z 330.9792 or any other significant PFK fragment in the range of 300 to 350.

9.4.2 Procedure for HRMS System Tune

9.4.2.1 Using a PFK molecular leak, tune the instrument to meet the minimum requirement in Section 9.4.1.2. For each descriptor (Table 7), monitor and record the resolution and exact m/z ratios of 3 to 5 reference peaks covering the mass range of the descriptor.

9.5 Ion Abundance Ratios, Minimum Levels, Signal-to-Noise (S/N) Ratios, and Window-Defining Mixture (WDM)

Choose an injection volume of either 1 or 2 µL, consistent with the capability of the HRGC/HRMS instrument. Inject a 1 or 2 µL aliquot of the calibration solution of lowest concentration, normally CS1 but CS0.2 can be used for more sensitive instruments (Table 5), using the GC conditions in Section 9.1.1.

9.5.1 Measure the SICP areas for each congener or congener group, and compute the ion abundance ratios at the exact m/z ratios specified in Table 7. Compare the computed ratio to the theoretical ratio given in Table 8.

9.5.1.1 The exact m/z ratios to be monitored in each descriptor are shown in Table 7. Each group or descriptor must be monitored in succession, as a function of GC RT, to ensure that the CB congeners of interest are detected. Additional m/z ratios may be monitored in each descriptor, and the m/z ratios may be divided among more than the descriptors listed in Table 7, provided that the laboratory is able to monitor the m/z ratios of all CB congeners that may be eluted from the GC in a given LOC window. The laboratory must also monitor exact m/z ratios for congeners at higher LOC to determine if fragments will compromise measurement of congeners at lower LOC.

9.5.1.2 The MS must be operated in a mass-drift correction mode, using PFK to provide lock m/z ratios. The lock mass for each group of m/z ratios is shown in Table 7. Each lock mass must be monitored and must not vary by more than ±20% throughout its respective RT window. Variations of lock mass by more than 20% indicate the presence of coeluting interferences that raise the source pressure and may significantly reduce the sensitivity of the MS. Reinjection of another aliquot of the sample extract may not resolve the problem and additional cleanup of the extract may be required to remove the interference. A lock mass interference or suppression in a RT region in which CB congeners and labeled compounds do not elute may be ignored.

9.5.2 All CB congeners and labeled compounds in the CS1 Standard must be within the Quality Control (QC) limits (Table 8) for their respective ion abundance ratios; otherwise, the MS must be adjusted and the test repeated until the m/z ratios fall within the limits specified. If the adjustment alters the resolution of the MS, resolution must be verified (Section 9.2.1) before a repeat of the test.

9.5.3 The peaks representing the CB congeners and labeled compounds in the lowest concentration Calibration Standard must have signal-to-noise (S/N) ratios ≥10; otherwise, the MS must be adjusted and the test repeated until this requirement is met. If this requirement cannot be met using CS0.2 then CS1 must be used for the lowest concentration standard.

9.5.4 Frequency of Window-Defining Mixture (WDM)

9.5.4.1 The WDM must be analyzed as follows:

- After the HRMS PFK tune and before any initial calibration on each instrument and HRGC column used for analysis;
- Once at the beginning of each 12-hour period during which standards or samples are analyzed; and
- Whenever adjustments or instrument maintenance activities are performed that may affect RTs.

9.5.4.2 The 12-hour time period for the HRGC/HRMS system performing check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the WDM that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after 12 hours have elapsed according to the system clock.

9.5.5 Procedure for WDM

9.5.5.1 Analyze a 1 or 2 µL aliquot of the WDM (CS1 can be used before any initial calibration; CS3 can be used before any continuing calibration).

9.5.5.2 Adjust the descriptor switching times and the HRGC column conditions as needed to ensure that the isomers elute in the appropriate ion descriptors. Table 2 provides the elution order (first/last) of the window-defining compounds.

9.5.5.3 Technical Acceptance Criteria for WDM

Technical acceptance criteria for the WDM must be met before any standards, samples, QC samples, and required blanks are analyzed. Any analysis conducted when the technical acceptance criteria have not been met will require reanalysis at no additional cost to USEPA.

9.5.5.3.1 Corrective Action for WDM

9.5.5.3.1.1 If the technical acceptance criteria for the WDM are not met, the instrument must be adjusted and the test repeated or the HRGC column must be replaced.

9.6 Initial Calibration

9.6.1 Summary of Initial Calibration

9.6.1.1 Before the analysis of samples and blanks, and after the HRGC/HRMS system performance check criteria have been met, each HRGC/HRMS system must be calibrated with a minimum of five concentrations to determine instrument sensitivity and linearity of the HRGC/HRMS response for the Toxic congeners.

9.6.2 Calibrations by Isotope Dilution

Isotope dilution is used for a calibration of the Toxics/LOC CB Congeners. The reference compounds for each native compound and its labeled analog are as listed in Table 2. A five- or six-point calibration encompassing the concentration range is prepared for each native congener.

9.6.2.1 For the Toxics/LOC CB Congers determined by isotope dilution, the Relative Response (RR) (labeled to native) vs. concentration in the calibration solutions (Table 5) is computed over the calibration range according to the procedures described below. Five calibration points are employed for less-sensitive HRMS instruments; five or six-points may be employed for more sensitive instruments.

9.6.2.2 The response of each Toxics/LOC CB relative to its labeled analog is determined using the area responses of both the primary and secondary exact m/z ratios specified in Table 7, for each Calibration Standard, as follows:

EQ. 1 Relative Response

$$RR = \frac{(A1_n + A2_n)\ C_l}{(A1_l + A2_l)\ C_n}$$

Where,

$A1_n$ and $A2_n$	=	The areas of the primary and secondary m/z ratios for the CB congener.
$A1_l$ and $A2_l$	=	The areas of the primary and secondary m/z ratios for the labeled compound.
C_l	=	The concentration of the labeled compound in the Calibration Standard (Table 5).
C_n	=	The concentration of the native Compound Standard (Table 5).

9.6.2.3 To calibrate the analytical system by isotope dilution, inject Calibration Standards CS1 through CS5 (Section 7.10 and Table 5) for a less sensitive instrument or CS0.2 through CS5 for a more sensitive instrument. Use a volume identical to the volume chosen in Section 9.5 and the conditions in Section 9.1.1. Compute and store the RR for each native Toxics/LOC CB Congener at each concentration. Compute the mean RR ($\overline{RR}$) and the Percent Relative Standard Deviation (%RSD) of the five (or six) RRs.

9.6.3 Initial Calibration by Internal Standard

An Internal Standard calibration is applied to the determination of the native CB congeners for which a labeled compound is not available, to the determination of the Labeled Toxics/LOC/Window-Defining Congeners and Labeled Cleanup Congeners, and to the determination of the Labeled Internal Standards except for CB 178. The reference compound for each compound is listed in Table 2. For the native congeners (other than the native Toxics/LOC CB Congeners), calibration is performed at a single point using the diluted combined 209-Congener Standard Solutions. For the labeled compounds, a calibration is performed using data from the five (or six) points in the calibration for the native Toxics/LOC CB Congeners (Section 9.6.2).

9.6.3.1 Relative Response Factors (RRFs) — Internal Standard calibration requires the determination of RRFs defined by the following equation:

EQ. 2 Relative Response Factor

$$RRF = \frac{(A1_s + A2_s)\ C_{is}}{(A1_{is} + A2_{is})\ C_s}$$

Where,

$A1_s$ and $A2_s$ = The areas of the primary and secondary m/z ratios for the CB congener.

$A1_{is}$ and $A2_{is}$ = The areas of the primary and secondary m/z ratios for the Internal Standard.

C_{is} = The concentration of the Internal Standard (Table 5).

C_s = The concentration of the compound in the Calibration Standard (Table 5).

9.6.3.2 To single-concentration calibrate the analytical system for native CB congeners other than the native Toxics/LOC CB Congeners by Internal Standard, inject the diluted combined 209-Congener Standard Solutions (Section 7.10.2.2 and Table 3). Use a volume identical to the volume chosen in Section 10.3 and the conditions in Section 9.1.1.

9.6.3.3 Compute and store the RRF for all native CB congeners except the Native Toxics/LOC CB Congeners. Use the average response of the labeled compounds at each LOC as the quantitation reference, as shown in Table 2. For the combinations of isomeric congeners that coelute, compute a combined RRF for the coeluted group. For example, for CB Congener 122, the areas at the two exact m/z ratios for 104L, 105L, 114L, 123L, 118L, and 126L are summed and the total area is divided by 6 (because there are 6 congeners in the quantitation reference).

NOTE: All labeled congeners at each LOC are used as reference to reduce the effect of interference if a single congener is used as reference. Other quantitation references and procedures may be used if the results produced are as accurate as results produced by the quantitation references and procedures described in this Section

9.6.3.4 Compute and store the RRF for the labeled compounds, except CB 178. For the Labeled Toxics/LOC/Window-Defining Congeners and the Labeled Cleanup Standards, use the nearest eluted Labeled Internal Standard as the quantitation reference, as given in Table 2. The Labeled Internal Standards are referenced to CB 178, as shown in Table 2.

9.6.4 Frequency of Initial Calibration

9.6.4.1 Each HRGC/HRMS system must be calibrated prior to analysis of samples under the contract, whenever the Contractor takes corrective action that may change or affect the initial calibration criteria (e.g., ion source cleaning or repairs, column replacement, etc.), or if the calibration verification technical acceptance criteria are not met.

9.6.4.2 If time still remains in the 12-hour time period after meeting the technical acceptance for the initial calibration, samples may be analyzed. It is not necessary to analyze a Calibration Verification Standard within this 12-hour period if the Initial Calibration Standard that is the same concentration as the Calibration Verification Standard meets the technical acceptance criteria. Quantitation of all the samples and blank results are necessary against the 66 and the mean RRF (666) from the initial calibration.

9.6.5 Procedure for Initial Calibration

9.6.5.1 Inject a volume identical to the volume chosen in Section 9.5 and the conditions in Section 9.1.1. of each of the remaining Calibration Standards (CS0.2 or CS2) through CS5. This volume must be identical to the volume and conditions chosen for the HRGC/HRMS system performance check. If concentrations of all 209 congeners are required then inject a volume identical to the volume chosen in Section 9.5 and the conditions in Section 9.1.1 of the diluted combined 209-Congener Standard Solution.

9.6.5.2 Compute the RR and RRF for each native and labeled congener respectively at each concentration level.

9.6.5.3 Determine RTs, S/N ratios, and ion abundance ratios for all Calibration Standards.

9.6.6 Technical Acceptance criteria for Initial Calibration

9.6.6.1 All Initial Calibration Standards must be analyzed at the concentration and frequency described.

9.6.6.2 The ion ratios must fall within the limits specified in Table 8.

9.6.6.3 The S/N ratios for the HRGC/HRMS signal in every SICP must be $10.

9.6.6.4 The RTs must fall within the appropriate RT windows established by analysis of CS1.

9.6.6.5 The %RSD for the RR must be ±20% over the five- to six-point calibration.

9.6.7 Corrective Action for Initial Calibration

9.6.7.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to change columns, adjust the system, and recalibrate until all the technical acceptance criteria are met.

9.6.7.2 All initial calibrations' technical acceptance criteria must be met before any IPR, samples, or blanks are analyzed. Any analysis conducted when the technical acceptance criteria have not been met will require reanalysis at no additional cost to USEPA.

9.7 Continuing Calibration

9.7.1 Summary of Calibration Verification

9.7.1.1 Calibration verification consists of verification of the mid-point CS3 Standard RR and RRF.

9.7.2 Frequency of Calibration Verification

9.7.2.1 A CS3 Standard must be analyzed at the beginning of each 12-hour period during which sample data are collected, but after the HRMS system tune, as well as at the end of each 12-hour period. If required, the diluted combined 209-Congener Standard Solutions (Section 7.10.2.2) must also be analyzed at the beginning of each 12-hour period, but after the CS3. The CS3 Standard analyzed at the end of a 12-hour period may also be used as the beginning of the next 12-hour period.

9.7.3 Procedure for Calibration Verification

9.7.3.1 Inject 1 or 2 µL of the CS3 Calibration Standard and measure the SICP areas for the analytes and compute the ion abundance ratios at the exact m/z ratios. Compare the ratio to the theoretical ratio. Verify that the system meets the ion abundance ratios, the minimum S/N ratios, and RT criteria. Compute the concentrations of the Toxics/LOC CB Congeners based on the initial calibration.

9.7.3.2 For each required native, LOC, and labeled congener, compare the concentration with the calibration verification limit in Table 6.

9.7.4 Technical Acceptance Criteria for Calibration Verification

9.7.4.1 All congeners in the standard must be with must be within their respective ion abundance ratios.

9.7.4.2 The RRTs of the congeners in the standard will be within the limits defined in Table 2.

9.7.4.3 The peaks representing the congeners in the standard must have a S/N ratio greater than or equal to 10.

9.7.4.4 The concentration calculated for each congener in the standard must be within the limits described in Table 6.

9.7.5 Corrective Action for Calibration Verification

9.7.5.1 Calibration Verification technical acceptance criteria must be met before any samples or blanks are analyzed. Any analysis conducted when the technical acceptance criteria have not been met will require reanalysis at no additional cost to USEPA.

9.7.5.2 If the calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to change columns, adjust the system, and recalibrate. If recalibration is required, recalibration for the 209 congeners must also be performed.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amounts (less than 90% of the required amount is received to perform the analysis, the Contractor will contact the Sample Management Office (SMO) to apprise them of the problem. SMO will contact the Region for instructions. The Region will either require that no sample analyses be performed, or require that a reduced volume be used for sample analysis. No other changes in the analyses will be permitted. The Contractor will document the Region's decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multi-phase samples (e.g., a two-phase liquid sample, oily sludge/sandy soil samples) are received by the Contractor, the Contractor will contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze each phase separately. SMO will provide EPA Sample Numbers for the additional phases.
- Separate the phases and analyze one or more of the phases, but not all of the phases. SMO will provide EPA Sample Numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If not all phases are amenable to analysis (i.e., outside scope), the Region may require the Contractor to do any of the following:

- Separate the phase(s) and analyze the phase(s) that is amenable to analysis. SMO will provide EPA Sample Numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor will document the Region's decision in the SDG Narrative.

10.1.3 Aqueous Samples

10.1.3.1 Three procedures are provided for the extraction of Chlorinated Biphenyl (CB) congeners from aqueous samples:

- Solid phase extraction (SPE).
- Separatory funnel extraction (SFE).
- Continuous liquid-liquid extraction (CLLE).

10.1.3.1.1 Solid Phase Extraction

10.1.3.1.1.1 Disk Preparation

10.1.3.1.1.1.1 Remove the test tube from the suction flask. Place an SPE disk on the base of the filter holder and wet with methylene chloride. While holding a GMF 150 filter above the SPE disk with tweezers, wet the filter with methylene chloride and lay the filter on the SPE disk, making sure that air is not trapped between the filter and disk. Clamp the filter and SPE disk between the 1 L glass reservoir and the vacuum filtration flask.

10.1.3.1.1.1.2 Rinse the sides of the reservoir with approximately 15 mL of methylene chloride using a squeeze bottle or pipet. Apply the vacuum momentarily until a few drops appear at the drip tip. Release the vacuum and allow the filter/disk to soak for approximately 1 min. Apply vacuum and draw all of the methylene chloride through the filter/disk. Repeat the wash step with approximately 15 mL of acetone and allow the filter/disk to air dry.

10.1.3.1.1.2 Sample Extraction

10.1.3.1.1.2.1 Pre-wet the disk by adding approximately 20 mL of methanol to the reservoir. Pull most of the methanol through the filter/disk, retaining a layer of methanol approximately 2 mm thick on the filter. Do not allow the filter/disk to go dry from this point until the extraction is completed.

10.1.3.1.1.2.2 Add approximately 20 mL of reagent water to the reservoir and pull most through, leaving a layer approximately 2 mm thick on the filter/disk.

10.1.3.1.1.2.3 Measure out a 1 L sample aliquot into a clean graduated cylinder. To this, add 5 mL of methanol and 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12) and allow equilibration.

10.1.3.1.1.2.4 Add the sample to its respective reservoir and turn on the vacuum to begin the extraction. Rinse the graduated cylinder twice with 5 mL of reagent water and add these rinses to the reservoir. Adjust the vacuum to complete the extraction in no less than 10 min. For samples containing a high concentration of particles (suspended solids), the extraction time may be an hour or longer.

10.1.3.1.1.2.5 Before the entire sample has been pulled through the filter/disk, rinse the sides of the reservoir with small portions of reagent water.

10.1.3.1.1.2.6 Partially dry the filter/disk under a vacuum for approximately 3 min.

10.1.3.1.1.3 Elution of the Filter/Disk

10.1.3.1.1.3.1 Release the vacuum, remove the entire filter/disk/reservoir assembly from the vacuum flask, and empty the flask. Insert a test tube for eluant collection into the flask. The test tube should have sufficient capacity to contain the total volume of the elution solvent (approximately 50 mL) and should fit around the drip tip. The drip tip should protrude into the test tube to preclude loss of a sample from spattering when the vacuum is applied. Reassemble the filter/disk/reservoir assembly on the vacuum flask.

10.1.3.1.1.3.2 Wet the filter/disk with 4-5 mL of acetone. Allow the acetone to spread evenly across the disk and soak for 15-20 sec. Pull the acetone through the disk, releasing the vacuum when approximately 1 mm thickness remains on the filter.

10.1.3.1.1.3.3 Release the vacuum, remove the filter/disk/reservoir assembly, and remove the test tube containing the sample solution. Quantitatively transfer the solution to a 250 mL separatory funnel and proceed to Section 10.2 for back-extraction.

10.1.3.1.2 Separatory Funnel Extraction

10.1.3.1.2.1 Measure out a 1 L sample aliquot into a clean graduated cylinder. To this, add 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12) and allow equilibration. Pour the spiked sample into a 2 L separatory funnel. Rinse the graduated cylinder twice with 5 mL of reagent water and add these rinses to the separatory funnel.

10.1.3.1.2.2 Add 60 mL methylene chloride to the graduated cylinder to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting. Allow the organic layer to separate from the aqueous phase for a minimum of 10 min. If an emulsion forms that is more than one-third the volume of the solvent layer, then employ mechanical techniques to complete the phase separation (see Note below). Drain the methylene chloride extract through a solvent-rinsed glass funnel approximately one-half full of granular anhydrous sodium sulfate (Section 7.2.1) supported on clean glass-fiber paper into a solvent-rinsed concentration device.

NOTE: If an emulsion forms, the laboratory must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, uses of phase separation paper, centrifugation, uses of an ultrasonic bath with ice, addition of NaCl, or other physical methods.

10.1.3.1.2.3 Extract the water sample two more times with 60 mL portions of methylene chloride. Drain each portion through the sodium sulfate into the concentrator. After the third extraction, rinse the separatory funnel with at least 20 mL of methylene chloride, and drain this rinse through the sodium sulfate into the concentrator. Repeat this rinse at least twice. Set aside the funnel with sodium sulfate if the extract is to be combined with the extract from the particles.

10.1.3.1.2.4 Concentrate the extract using one of the macro-concentration procedures in Section 10.3.

10.1.3.1.3 Continuous Liquid-Liquid Extraction

10.1.3.1.3.1 Place 100 to 150 mL methylene chloride in each continuous extractor and 200-300 mL in each distilling flask.

10.1.3.1.3.2 Measure out a 1 L sample aliquot into a clean graduated cylinder. To this, add 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12) and allow equilibration. Pour the spiked sample into the extractor. Rinse the graduated cylinder with 50-100 mL of methylene chloride and add this rinse to the extractor.

10.1.3.1.3.3 Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1-2 drops of methylene chloride per second will fall from the condenser tip into the water. Extract the compound for 16-24 hours.

10.1.3.1.3.4 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7-10 cm of granular anhydrous sodium sulfate into a 500 mL Kuderna-Danish (K-D) evaporator flask equipped with a 10 mL concentrator tube. Rinse the distilling flask with 30 to 50 mL of methylene chloride and pour through the drying column. Concentrate and exchange to hexane per Section 10.4 and back-extract per Section 10.2.

10.1.4 Soil/Sediment Samples

Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

10.1.4.1 Percent Solid

Immediately after weighing each sample for extraction, weigh 5.00-10.0 g of the soil/sediment into a tared crucible. Determine the Percent Solid by drying overnight at 105°C. Allow cooling in a desiccator before weighing. Concentrations of individual toxic congeners will be reported relative to the dry weight of soil/sediment.

EQ 3. Calculation of Percent Solid

$$\%\ \text{Solid} = \frac{\text{grams of dry sample}}{\text{grams of wet sample}} \times 100$$

10.1.4.2 Soxhlet/Dean-Stark (SDS) Extraction

10.1.4.2.1 Charge a clean extraction thimble (Section 6.4.2.2) with 5.0 g of activated 100/200 mesh silica (Section 7.5.1.1) topped with 100 g of quartz sand (Section 7.3.2).

NOTE: Do not disturb the silica layer throughout the extraction process.

10.1.4.2.2 Place the thimble in a clean extractor. Place 30-40 mL of toluene in the receiver and 200-250 mL of toluene in the flask.

10.1.4.2.3 Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene will fall per second from the condenser tip into the receiver. Extract the apparatus for a minimum of 3 hours.

10.1.4.2.4 After pre-extraction, cool and disassemble the apparatus. Rinse the thimble with toluene and allow to air dry.

10.1.4.2.5 Weigh approximately 10 g of a sample, to the nearest 0.1 g, and spike with 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12). Transfer this into the thimble and manually mix into the sand layer with a clean metal spatula, carefully breaking up any large lumps of the sample.

10.1.4.2.6 Reassemble the pre-extracted SDS apparatus, and add a fresh charge of toluene to the receiver and reflux flask. Apply power to the heating mantle to begin refluxing. Adjust the reflux rate to match the rate of percolation through the sand and silica beds until water removal lessens the restriction to toluene flow. Frequently check the apparatus for foaming during the first 2 hours of extraction. If foaming occurs, reduce the reflux rate until foaming subsides.

10.1.4.2.7 Drain the water from the receiver at 1-2 hours and 8-9 hours, or sooner if the receiver fills with water. Reflux the sample for a total of 16-24 hours. Cool and disassemble the apparatus. Record the total volume of water collected.

10.1.4.2.8 Remove the distilling flask. Drain the water from the Dean-Stark receiver and add any toluene in the receiver to the extract in the flask.

10.1.4.2.9 Concentrate the extracts from particles to approximately 10 mL using the rotary evaporator (Section 10.3.1) or heating mantle (Section 10.3.2), transfer to a 250 mL separatory funnel, and proceed with back-extraction (Section 10.2).

10.1.5 Tissue Samples

10.1.5.1 Homogenization

Before processing tissue samples, the laboratory must determine the exact tissue to be analyzed. Common requests for analysis of fish tissue include whole fish-skin on, whole fish-skin removed, edible fish fillets (filleted in the field or by the laboratory), specific organs, and other portions. Once the appropriate tissue has been determined, the sample must be homogenized.

10.1.5.1.1 Samples are homogenized while still frozen, where practical. If the laboratory must dissect the whole fish to obtain the appropriate tissue for analysis, the unused tissues may be rapidly re-frozen and stored in a clean glass jar for subsequent use.

10.1.5.1.2 Each analysis requires 10 g of tissue (wet weight). Therefore, the laboratory should homogenize at least 20 g of tissue to allow for reextraction of a second aliquot of the same homogenized sample, if reanalysis is required. When whole fish analysis is necessary, the entire fish is homogenized.

10.1.5.1.3 Homogenize the sample in a tissue homogenizer (Section 6.2.3) or grind in a meat grinder (Section 6.2.4). Cut tissue too large to feed into the grinder into smaller pieces. To assure homogeneity, grind three times.

10.1.5.1.4 Transfer approximately 10 g (wet weight) of homogenized tissue to a clean, tared, 400-500 mL beaker.

10.1.5.1.5 Transfer the remaining homogenized tissue to a clean jar with a polytetrafluoroethylene (PTFE)-lined lid. Seal the jar and store the tissue at less than -10°C. Return any tissue that was not homogenized to its original container and store at less than -10°C.

10.1.5.2 Soxhlet Extraction

10.1.5.2.1 Spike 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12) into the sample.

10.1.5.2.2 Add 30 to 40 g of powdered anhydrous sodium sulfate (Section 7.2.2) to each of the beakers and mix thoroughly. Cover the beakers with aluminum foil and allow equilibration for 12-24 hours. Remix before extraction to prevent clumping.

10.1.5.2.3 Assemble and pre-extract the Soxhlet apparatus per Sections 10.1.4.2.3 - 10.1.4.2.4, except use the (1:1) (v/v) hexane/methylene chloride mixture for the pre-extraction and rinsing and omit the quartz sand.

10.1.5.2.4 Reassemble the pre-extracted Soxhlet apparatus and add a fresh charge of (1:1) (v/v) hexane/methylene chloride to the reflux flask.

10.1.5.2.5 Transfer the sample/sodium sulfate mixture (Section 10.1.5.2.2) to the SDS thimble, and install the thimble in the Soxhlet apparatus.

10.1.5.2.6 Rinse the beaker with several portions of solvent mixture and add to the thimble. Fill the thimble/receiver with solvent. The extract process takes 18-24 hours.

10.1.5.2.7 After extraction, cool and disassemble the apparatus.

10.1.5.2.8 Quantitatively transfer the extract to a macro-concentration device (Section 10.3) and concentrate to near dryness. Set aside the concentration apparatus for reuse.

10.1.5.2.9 Complete the removal of the solvent using the nitrogen blowdown procedure (Section 10.4) and a water bath temperature of 60°C. Weigh the receiver, record the weight, and return the receiver to the blowdown apparatus, concentrating the residue until a constant weight is obtained.

10.1.5.3 Percent Lipids

10.1.5.3.1 Re-dissolve the residue from Section 10.1.5.2.9 in the receiver in hexane and spike 1.00 mL of the Labeled Cleanup Standard Spiking Solution (Section 7.13) into the solution.

10.1.5.3.2 Transfer the residue/hexane to the anthropogenic isolation column (Section 10.5.5.2), retaining the boiling chips in the concentration apparatus. Use several rinses to assure that all material is transferred. If necessary, sonicate or heat the receiver slightly to assure that all material is re-dissolved. Allow the receiver to dry. Weigh the receiver and boiling chips to three significant figures.

10.1.5.3.3 Calculate the lipid content to the nearest three significant figures as follows:

EQ. 4 Percent Lipid Determination

$$\text{Percent Lipid} = \frac{\text{Weight of residue (g)}}{\text{Weight of tissue (g)}} \times 100$$

10.1.5.3.4 The laboratory should determine the lipid content of the blank to assure that the extraction system is working effectively.

10.2 Back-Extraction with Base and Acid

10.2.1 Back-extraction is applied to extracts from aqueous and soil/sediment samples. Back-extraction is applied directly to Solid phase extraction extracts. Back-extraction is applied to extracts from the separatory funnel, CLLE, and SDS procedures after macro-concentration (Section 10.3) of the extract. Back-extraction may not be necessary for some samples. For some samples, the presence of color in the extract may indicate that back-extraction is necessary. If back-extraction is not performed, spike 1 mL of the Labeled Cleanup Standard Spiking Solution (Section 7.13) into the extract and concentrate the extract for cleanup or analysis (Section 10.4). If back-extraction is necessary, spike 1 mL of the Labeled Cleanup Standard Spiking Solution (Section 7.13) into the extracts and proceed with macro-concentration.

10.2.2 Transfer the (concentrated) extract to a 250 mL separatory funnel. Rinse the concentration vessel with small portions of hexane, adjust the hexane volume in the separatory funnel to 10-20 mL and proceed to back-extraction. Partition the extract against 50 mL of potassium hydroxide solution (Section 7.1.1). Shake for 2 min. with periodic venting into a hood. Remove and discard the aqueous layer. Repeat the base washing until no color is visible in the aqueous layer, to a maximum of four washes. Minimize contact time between the extract and the base to prevent degradation of the CB congeners. Stronger potassium hydroxide solutions may be used for back-extraction.

10.2.3 Partition the extract against 50 mL of sodium chloride solution (Section 7.1.4) in the same way as with base. Discard the aqueous layer.

10.2.4 Partition the extract against 50 mL of sulfuric acid (Section 7.1.2) in the same way as with base. Repeat the acid washing until no color is visible in the aqueous layer, to a maximum of four washes.

10.2.5 Repeat the partitioning against sodium chloride solution and discard the aqueous layer.

10.2.6 Pour each extract through a drying column containing 7 to 10 cm of granular anhydrous sodium sulfate (Section 7.2.1). Rinse the separatory funnel with 30 to 50 mL of solvent, and pour through the drying column. Collect each extract in a round-bottom flask. Reconcentrate the sample Sections 10.3 - 10.4, and cleanup the samples per Section 10.5.

10.3 Macro-Concentration

Extracts in toluene are concentrated using a rotary evaporator or a heating mantle; extracts in methylene chloride or hexane are concentrated using a rotary evaporator, heating mantle, or Kuderna-Danish apparatus.

NOTE: In the concentration procedures below, the extract must not be allowed to concentrate to dryness because the mono- through tri-chlorobiphenyls may be totally or partially lost.

10.3.1 Rotary Evaporation - Concentrate the extracts in separate round-bottom flasks.

10.3.1.1 Assemble the rotary evaporator according to manufacturers' instructions, and warm the water bath to 45°C. On a daily basis, pre-clean the rotary evaporator by concentrating 100 mL of clean extraction solvent through the system. Between samples, three 2-3 mL aliquots of solvent should be rinsed down the feed tube into a waste beaker.

10.3.1.2 Attach the round-bottom flask containing the sample extract to the rotary evaporator. Slowly apply a vacuum to the system, and begin rotating the sample flask.

10.3.1.3 Lower the flask into the water bath, and adjust the speed of rotation and the temperature as required to complete concentration in 15-20 min. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

10.3.1.4 When the liquid in the concentration flask has reached an apparent volume of approximately 2 mL, remove the flask from the water bath and stop the rotation. Slowly and carefully, admit air into the system. Be sure not to open the valve so quickly that the sample is blown out of the flask. Rinse the feed tube with approximately 2 mL of solvent.

10.3.1.5 The extract is now ready for back-extraction, or micro-concentration and solvent exchange.

10.3.2 Heating Mantle - Concentrate the extracts in separate round-bottom flasks.

10.3.2.1 Add 1 or 2 clean boiling chips to the round-bottom flask, and attach a three-ball macro Snyder column. Pre-wet the column by adding approximately 1 mL of solvent through the top. Place the round-bottom flask in a heating mantle and apply heat as required to complete the concentration in 15-20 min. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

10.3.2.2 When the liquid has reached an apparent volume of approximately 10 mL, remove the round-bottom flask from the heating mantle and allow the solvent to drain and cool for at least 10 min. Remove the Snyder column and rinse the glass joint into the receiver with small portions of the solvent.

10.3.2.3 The extract is now ready for back-extraction, or micro-concentration and solvent exchange.

10.3.3 Kuderna-Danish (K-D) - Concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes. The K-D technique is used for solvents such as methylene chloride and hexane. Toluene is difficult to concentrate using the K-D technique unless a water bath fed by a steam generator is used.

10.3.3.1 Add 1 or 2 clean boiling chips to the receiver. Attach a three-ball macro Snyder column. Pre-wet the column by adding approximately 1 mL of solvent through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam.

10.3.3.2 Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 min. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

10.3.3.3 When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent draining and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of solvent. A 5 mL syringe is recommended for this operation.

10.3.3.4 Remove the three-ball Snyder column, add a fresh boiling chip, and attach a two-ball micro Snyder column to the concentrator tube. Pre-wet the column by adding approximately 0.5 mL of solvent through the top. Place the apparatus in the hot water bath.

10.3.3.5 Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 min. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

10.3.3.6 When the liquids reach an apparent volume of 0.5 mL, remove the apparatus from the water bath and allow it to drain and cool for at least 10 min.

10.3.3.7 The extract is now ready for back-extraction, or micro-concentration and solvent exchange.

10.4 Micro-Concentration and Solvent Exchange

10.4.1 Transfer the extracts from Section 10.3 to a blowdown vial using 2-3 rinses of solvent.

10.4.2 Extracts are to be subjected to Gel Permeation Chromatography (GPC) Cleanup are exchanged into methylene chloride. Extracts to be cleaned up using silica gel, carbon, Florisil, and/or HPLC are exchanged into hexane.

10.4.3 Transfer the vial containing the sample extract to a nitrogen blowdown device. Adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed.

NOTE: A large vortex in the solvent may cause analyte loss.

10.4.4 Lower the vial into a 45°C water bath and continue concentrating.

10.4.4.1 If the extract or an aliquot of the extract is to be concentrated to dryness for weight determination (Sections 10.1.5.2.9), blow-dry until a constant weight is obtained.

10.4.4.2 If the extract is to be concentrated for injection into the Gas Chromatograph/Mass Spectrometer (GC/MS) or the solvent is to be exchanged for an extract cleanup, proceed as follows:

10.4.4.2.1 When the volume of the liquid is approximately 100 µL, add 2-3 mL of the desired solvent (methylene chloride for GPC and HPLC, or hexane for the other cleanups) and continue concentration to approximately 100 µL, repeat the addition of solvent and concentrate once more.

10.4.4.2.2 If the extract is to be cleaned up by GPC, adjust the volume of the extract to 5.0 mL with methylene chloride. If the extract is to be cleaned up by HPLC, concentrate the extract to 1.0 mL. Proceed with GPC or HPLC Cleanup (Sections 10.5.1 and 10.5.4).

10.4.4.2.3 If the extract is to be cleaned up by column chromatography (silica gel, Carbopak/Celite, or Florisil), bring the final volume to 1.0 mL with hexane. Proceed with a column cleanup.

10.4.4.2.4 If the extract is to be concentrated for injection into the GC/MS (Section 10.6.3), quantitatively transfer the extract to a 0.3 mL conical vial for final concentration, rinsing the larger vial with hexane and adding the rinse to the conical vial. Reduce the volume to approximately 100 µL. Add 20 µL of nonane to the vial, and evaporate the solvent to 20 µL. Seal the vial and label with the Sample Number. Store in the dark at room temperature until ready for GC/MS analysis. If GC/MS analysis will not be performed on the same day, store the vial at less than -10°C.

10.5 Sample Cleanup

The cleanup may not be necessary for relatively clean samples (e.g., treated effluents, groundwater, and drinking water). If particular circumstances require the use of cleanup procedure, the laboratory may use any or all of the procedures below.

10.5.1 Gel Permeation Chromatography (GPC)

GPC removes high molecular weight interferences that cause GC column performance to degrade. It should be used for all soil and sediment extracts. It may be used for water extracts that are expected to contain high molecular weight organic compounds (e.g., polymeric materials and humic acids). It should also be used for tissue extracts after an initial cleanup on the anthropogenic isolation column (Section 10.5.5).

10.5.1.1 Prepare the GPC column by placing 70 to 75 g of SX-3 Bio-beads (Section 6.6.1.1) in a 400-500 mL beaker. Cover the beads with methylene chloride and allow swelling overnight (a minimum of 12 hours). Transfer the swelled beads to the column (Section 6.6.1.1) and pump solvent through the column, from bottom to top, at 4.5 - 5.5 mL/min before connecting the column to the detector. After purging the column with solvent for 1 to 2 hours, adjust the column head pressure to 7 to 10 psig and purge for 4 to 5 hours to remove air. Maintain a head pressure of 7-10 psig. Connect the column to the detector (Section 10.5.5).

10.5.1.2 Calibrate the column by loading 5.0 mL of the GPC calibration solution (Section 7.4) into the sample loop. Inject the GPC calibration solution and record the signal from the detector. The elution pattern will be corn oil, BEHP, methoxychlor, perylene, and sulfur. Set the "dump time" to allow greater than 85% removal of BEHP and greater than 85% collection of methoxychlor. Set the "collect time" to the time of the sulfur peak maximum. Verify the calibration with the GPC calibration solution after every 20 extracts. The calibration is verified if the recovery of the methoxychlor is greater than 85%. If calibrations are not verified, the system must be recalibrated using the GPC calibration solution, and the previous sample batch must be reextracted and cleaned up using the calibrated GPC system.

10.5.1.3 Filter the extract or load through the filter holder (Section 6.6.1.3) to remove particles. Load the 5.0 mL extract onto the column. Elute the extract using the calibration data determined in Section 10.5.1.2. Collect the eluate in a clean 400-500 mL beaker. Allow the system to rinse for an additional 10 min. before injecting the next sample. Rinse the sample loading tube thoroughly with methylene chloride between extracts to prepare for the next sample. If an extract is encountered that could overload the GPC column to the extent that carry-over could occur, a 5.0 mL methylene chloride blank must be run through the system to check for carry-over. Concentrate the eluate per Sections 10.3 and 10.4 for further cleanup or injection into the GC/MS.

10.5.2 Silica Gel (acid, neutral, and base) cleanup is used to remove non-polar and polar interferences.

10.5.2.1 Place a glass wool plug in a 15 mm ID chromatography column (Section 6.6.4.2). Pack the column bottom to top with: 1 g silica gel (Section 7.5.1.1), 4 g basic silica gel (Section 7.5.1.3), 1 g silica gel, 8 g acidic silica gel (Section 7.5.1.2), 2 g silica gel, and 4 g granular anhydrous sodium sulfate (Section 7.2.1). Tap the column to settle the adsorbent. Column packing material may be slurried in hexane to aid settling.

10.5.2.2 Pre-elute the column with 50-100 mL of hexane. Close the stopcock when the hexane is within 1 mm of the sodium sulfate. Discard the eluate. Check the column for channeling. If channeling is present, discard the column and prepare another.

10.5.2.3 Apply the concentrated extract to the column. Open the stopcock until the extract is within 1 mm of the sodium sulfate.

10.5.2.4 Rinse the receiver twice with 1 mL portions of hexane, and apply separately to the column. Elute the CB congeners with 25 mL of hexane and collect the eluate.

10.5.2.5 Concentrate the eluate (per Sections 10.3 and 10.4) for further cleanup or injection into the HPLC or GC/MS.

10.5.2.6 For extracts of samples known to contain large quantities of other organic compounds, it may be advisable to increase the capacity of the silica gel column. This may be accomplished by increasing the strengths of the acidic and basic silica gels. The acidic silica gel (Section 7.5.1.2) may be increased in strength to as much as 40% w/w (6.7 g sulfuric acid added to 10 g silica gel). The basic silica gel (Section 7.5.1.3) may be increased in strength to as much as 33% w/w (50 mL 1N NaOH added to 100 g silica gel), or the potassium silicate (Section 7.5.1.4) may be used.

NOTE: The use of stronger acidic silica gel (44% w/w) may lead to charring of organic compounds in some extracts. The charred material may retain some of the analytes and lead to lower recoveries of the CB congeners. Increasing the strengths of the acid and basic silica gel may also require increased volumes of hexane than those specified above to elute the analytes from the column.

10.5.3 The carbon column's cleanup can be used to separate Congeners 77, 126, and 169 from the mono- and di-ortho-substituted CB congeners.

10.5.3.1 Cut both ends from a 50 mL disposable serological pipet (Section 6.6.3.2) to produce a 20 cm column. Fire-polish both ends and flare both ends if desired. Insert a glass wool plug at one end, and pack the column with 3.6 g of Carbopak/Celite (Section 7.5.2.3) to form an adsorbent bed 20 cm long. Insert a glass wool plug on top of the bed to hold the adsorbent in place.

10.5.3.2 Pre-elute the column with 20 mL each in succession of toluene, methylene chloride, and hexane.

10.5.3.3 When the solvent is within 1 mm of the column packing, apply the n-hexane sample extract to the column. Rinse the sample container twice with 1 mL portions of hexane and apply separately to the column. Apply 2 mL of hexane to complete the transfer.

10.5.3.4 Elute the column with 25 mL of n-hexane and collect the eluate. This fraction will contain the mono- and di-ortho CB congeners. If carbon particles are present in the eluate, filter through glass-fiber filter paper.

10.5.3.5 Elute the column with 15 mL of methanol and discard the eluate. The fraction discarded will contain residual lipids and other potential interferents, if present.

10.5.3.6 Elute the column with 15 mL of toluene and collect the eluate. This fraction will contain Congeners 77, 126, and 169. If carbon particles are present in the eluate, filter through glass-fiber filter paper.

10.5.3.7 Concentrate the fractions per Section 10.3 and 10.4 for further cleanup or injection into the HPLC or GC/MS.

10.5.4 HPLC is used to provide specificity for certain congeners and congener groups.

10.5.4.1 Column Calibration

10.5.4.1.1 Prepare a Calibration Standard containing the Toxics and other congeners of interest at the concentrations of the stock solution in Table 3, or at a concentration appropriate to the response (not exceeding the linearity) of the detector.

10.5.4.1.2 Inject the Calibration Standard into the HPLC and record the signal from the detector. Collect the eluant for reuse. Elution will be in the order of the di-ortho, mono-ortho, and non-ortho congeners.

10.5.4.1.3 Establish the collection time for the congeners of interest. Following calibrations, flush the injection system with solvent to ensure that residual CB congeners is removed from the system.

10.5.4.1.4 Verify the calibration with the calibration solution after every 20 extracts. The calibration is verified if the recovery of the CB congeners is 75-125% compared to the initial calibration. If calibrations are not verified, the system must be recalibrated using the calibration solution, and the previous 20 samples must be reextracted and cleaned up using the calibrated system.

10.5.4.2 Extract Cleanup

10.5.4.2.1 Rinse the sides of the vial containing the sample and adjust to the volume required for the sample loop for injection.

10.5.4.2.2 Inject the sample extract into the HPLC.

10.5.4.2.3 Elute the extract using the calibration data determined in Section 10.5.4.1. Collect the fraction(s) in clean 20 mL concentrator tubes.

10.5.4.2.4 If an extract containing greater than 500 µg of total CB congeners is encountered, a blank must be run through the system to check for carry-over.

10.5.4.2.5 Concentrate the eluate per Section 10.4 for injection into the GC/MS.

10.5.5 Anthropogenic Isolation Column Cleanup is used for removal of lipids from tissue samples.

10.5.5.1 Prepare the column as given in Section 7.5.3.

10.5.5.2 Pre-elute the column with 100 mL of hexane. Drain the hexane layer to the top of the column, but do not expose the sodium sulfate.

10.5.5.3 Load the sample and rinses (Section 10.1.5.3.2) onto the column by draining each portion to the top of the bed. Elute the CB congeners from the column into the apparatus used for concentration (Section 10.3) using 200 mL of hexane.

10.5.5.4 Remove a small portion (e.g., 50 µL) of the extract for determination of residue content. Estimate the percent of the total that this portion represents. Concentrate the small portion to constant weight per Section 10.4.4.1. Calculate the total amount of residue in the extract. If more than 500 mg of material remains, repeat the cleanup using a fresh anthropogenic isolation column.

10.5.5.5 If necessary, exchange the extract to a solvent suitable for the additional cleanups to be used. GPC (Section 10.5.1) and Florisil (Section 10.5.6) are recommended as minimum additional cleanup steps.

10.5.5.6 Following cleanups, concentrate the extract to 20 µL as described in Section 10.4 and proceed with the analysis.

10.5.6 Florisil cleanup is used to remove non-polar and polar interferences.

10.5.6.1 Begin draining the n-hexane from the column (Section 7.5.4.1.2). Adjust the flow rate of eluant to 4.5 - 5.0 mL/min.

10.5.6.2 When the n-hexane is within 1 mm of the sodium sulfate, apply the sample extract (in hexane) to the column as close to the packing material as possible. Rinse the sample container twice with 1 mL portions of hexane and apply to the column.

10.5.6.3 Elute the mono-ortho and di-ortho CB congeners with approximately 165 mL of n-hexane and collect the eluate. Elute the non-ortho co-planar CB congeners with approximately 100 mL of 6% ether/hexane and collect the eluate. The exact volumes of solvents will need to be determined for each batch of Florisil. If the mono/di-ortho CB congeners is not to be separated from the non-ortho co-planar CB congeners, elute all CB congeners with 6% ether/hexane.

10.5.6.4 Concentrate the eluate(s) per Sections 10.3 - 10.4 for further cleanups or for injection into the HPLC or GC/MS.

10.6 Sample Analysis by HRGC/HRMS

10.6.1 Sample extracts will be analyzed only after the HRGC/HRMS system has met the resolution, Retention Time (RT), Relative Retention Time (RRT), and ion abundance ratio requirements in Section 10 and has met the requirements for initial calibration and Continuing Calibration Verification (CCV). The same instrument conditions must be employed for the analysis of samples as were used for a calibration.

10.6.2 Add 2 µL of the labeled Internal Standard Spiking Solution (Section 7.14) to the 20 µL sample extract immediately before injection to minimize the possibility of loss by evaporation, adsorption, or reaction. If an extract is to be reanalyzed and evaporation has occurred, do not add more Labeled Internal Standard Spiking Solution. Rather, bring the extract back to its previous volume (e.g., 19 µL) with pure nonane (18 µL if 2.0 µL injections are used).

10.6.3 Inject 1.0 or 2.0 µL of the concentrated extract containing the Labeled Internal Standards using on-column or splitless injection. The volume injected must be identical to the volume used for the calibration (Section 9). Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Monitor the exact m/z ratios at each LOC throughout the LOC RT window. If necessary, monitor m/z ratios associated with congeners at a higher LOC to assure that fragments are not interfering with the m/z ratios for congeners at a lower LOC. Also where warranted, monitor m/z ratios associated with interferents expected to be present. Stop data collection after $^{13}C_{12}$-DeCB has eluted. Return the column to the initial temperature for analysis of the next extract or standard.

10.6.4 Analysis of Complex Samples

Some samples may contain high levels (greater than 10 ng/L; greater than 1000 ng/kg) of the compounds of interest, interfering compounds, and/or polymeric materials. Some extracts may not concentrate to 20 µL (Section 10.4); others may overload the GC column and/or MS. Fragment ions from congeners at higher LOC may interfere with determination of congeners at a lower LOC. Analyze a smaller aliquot of the sample (Section 11.2.2.5) when the extract will not concentrate to 20 µL after all cleanup procedures have been exhausted. If a smaller aliquot of soils or mixed-phase samples is analyzed, assure that the aliquot is representative of the sample as a whole.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

A Chlorinated Biphenyl (CB) or labeled compound is identified in a standard, blank, or sample when all of the criteria in Sections 11.1 are met.

11.1.1 The signals for the two exact m/z ratios in Table 7 must be present and must maximize within the same two scans.

11.1.2 The signal-to-noise (S/N) ratio for the analyte peak at each exact m/z must be greater than or equal to 2.5 for each CB detected in a sample extract, and greater than or equal to 10 for all CB congeners in the Calibration Verification Standards (Sections 9.5.3).

11.1.3 The ratio of the integrated areas of the two exact m/z ratios specified in Table 7 must be within the limits in Table 8.

11.1.4 The Relative Retention Time (RRT) of the peak for a CB must be within the RRT QC limits specified in Table 2 or, if an alternate column or column system is employed, within its respective RRT Quality Control (QC) limits for the alternate column or column system (Section 6.8).

NOTE: For native CB congeners determined by Internal Standard quantitation, a given CB congener may fall within more than one Retention Time (RT) window and be mis-identified unless the RRT windows are made very narrow, as in Table 2. Therefore, consistency of the RT and RRT with other congeners and the labeled compounds may be required for rigorous congener identification. RT regression analysis may aid in this identification.

11.1.5 Because of congener overlap and the potential for interfering substances, it is possible that not all of the identification criteria may be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same RT. If identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the congener.

11.1.6 Technical Acceptance Criteria for Qualitative Identification

11.1.6.1 If the criteria for identification in Sections 11.1.1 to 11.1.5 are not met, the CB has not been identified and the result for that congener may not be reported. If interferences preclude identification, a new aliquot of the sample must be extracted, further cleaned up, and analyzed.

11.2 Quantitative Determination

11.2.1 Isotope Dilution Quantitation

11.2.1.1 By adding a known amount of the Labeled Toxics/Level of Chlorination (LOC)/Window-Defining Congeners to every sample before extraction, correction for recovery of the CB can be made because the native compound and its labeled analog exhibit similar effects upon extraction, concentration, and Gas Chromatography (GC). Relative Responses (RRs) are used in conjunction with the calibration data in Section 9 to determine concentrations in the final extract, so long as labeled compound spiking levels are constant.

11.2.1.2 Compute the concentrations in the extract of the native Toxics/LOC CB Congeners using the RRs from the calibration data (Section 9) and the following equation:

EQ. 5 Native Toxics Extract Concentration

$$C_{ex}\ (\text{ng/mL}) = \frac{(A1_n + A2_n)\ C_l}{(A1_l + A2_l)\ RR}$$

Where,

C_{ex} = The concentration of the CB in the extract. The other terms are defined in Section 9.6.2.2.

11.2.2 Internal Standard Quantitation and Labeled Compound Recovery

11.2.2.1 Compute the concentrations in the extract of the native compounds other than those in the native Toxics/LOC Standard, in the Labeled Cleanup Standard, and in the Labeled Internal Standard (except for Labeled CB 178) using the Relative Response Factors (RRF) determined from the calibration data (Section 9) and the following equation:

EQ. 6 Labeled CB Congeners and Non-Toxic Native Extract Concentration

$$C_{ex}\ (\text{ng/mL}) = \frac{(A1_s + A2_s)\ C_{is}}{(A1_{is} + A2_{is})\ RRF}$$

Where,

C_{ex} = The concentration of the labeled compound in the extract. The other terms are defined in Section 9.6.3.1.

11.2.2.2 Using the concentration in the extract determined above, compute the Percent Recovery (%R) of the Labeled Toxics/LOC/Window-Defining CB Congeners and the Labeled Cleanup Standard CB Congeners using the following equation:

EQ. 7 Percent Recovery Determination

$$\text{Recovery (\%)} = \frac{\text{Concentration found } (\mu\text{g/mL})}{\text{Concentration spiked } (\mu\text{g/mL})} \times 100$$

11.2.2.3 The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids (Section 10), as follows:

EQ. 8 Solid Sample Concentration

$$\text{Concentration in Aqueous Phase (pg/L)} = \frac{(C_{ex} \times V_{ex} \times D_f \times S)}{V_s}$$

Where,

C_{ex} = The concentration of the compound in the extract.

V_{ex} = The extract volume in mL.

W_s = The sample weight in kg.

S = Percent Solids Factor (weight of dry sample/weight of wet sample).

DF = Dilution Factor. The DF is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

11.2.2.4 The concentration of a native CB in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted (Section 11.4), as follows:

EQ. 9 Aqueous Sample Concentration

$$\text{Concentration in Aqueous Phase (pg/L)} = 1000 \times \frac{(C_{ex} \times V_{ex} \times D_f)}{V_s}$$

Where,

C_{ex} = The concentration of the compound in the extract.

V_{ex} = The extract volume in mL.

V_s = The sample volume in L.

D_f = Same as Eq. 8.

11.2.2.5 If the Selected Ion Current Profile (SICP) area at either quantitation m/z for any congener exceeds the calibration range of the system, dilute the sample extract by the factor necessary to bring the concentration within the calibration range, adjust the concentration of the Labeled Internal Standard to 100 pg/µL in the extract, and analyze an aliquot of this diluted extract. If the CB congeners cannot be measured reliably by isotope dilution, dilute and analyze an aqueous sample or analyze a smaller portion of a soil, tissue, or mixed-phase sample. Adjust the CB congener concentrations, detection limits, and minimum levels to account for the dilution.

12.0 QUALITY CONTROL (QC)

12.1 Continuing Calibration

At the beginning of each 12-hour shift during which analyses are performed, High Resolution Gas Chromatograph/High Resolution Mass Spectrometer (HRGC/HRMS) system performance and calibration are verified for all native Chlorinated Biphenyl (CB) congeners and labeled compounds. For these tests, analysis of the CS3 Calibration Verification Standard (Section 7.10.1 and Table 5) and the diluted combined 209-Congener Standard Solution (Section 7.10.2.2 and Table 5) must be used to verify all performance criteria. Adjustment and/or recalibration (Section 9) must be performed until all performance criteria are met. Only after all performance criteria are met may samples and blanks be analyzed.

12.1.1 Mass Spectrometer (MS) Resolution

Static resolving power checks must be performed at the beginning and at the end of each shift per Sections 9. If analyses are performed on successive shifts, only the beginning of the shift static resolving power check is required. If the requirement in Section 9 cannot be met, the problem must be corrected before analyses can proceed. If any of the samples in the previous shift may be affected by poor resolution, those samples must be reanalyzed at no additional cost to USEPA.

12.1.2 Calibration Verification

12.1.2.1 Inject the CS3 Standard using the procedure in Section 10.6.

12.1.2.2 The m/z abundance ratios for all CB congeners must be within the limits in Table 8; otherwise, the MS must be adjusted until the m/z abundance ratios fall within the limits specified when the verification test is be repeated. If the adjustment alters the resolution of the MS, resolution must be verified (Section 9) before repeating the verification test.

12.1.2.3 The chromatogram peak representing each native CB and labeled compounds in the CS3 Standard must be present with a S/N ratio of at least 10; otherwise, the MS must be adjusted and the verification test repeated.

12.1.2.4 Compute the concentration of the Toxics/LOC CB congeners by isotope dilution (Section 11.2). These concentrations are computed based on the calibration data in Section 9.

12.1.2.5 Technical Acceptance Criteria for Calibration Verification

For each compound, compare the concentration with the calibration verification limit in Table 6. If all compounds meet the acceptance criteria, a calibration has been verified and analysis of standards and sample extracts may proceed. If, however, any compound fails its respective limit, the measurement system is not performing properly. In this event, prepare a fresh Calibration Standard or correct the problem and repeat the resolution (Section 12.1.1) and verification (Section 12.1.2) tests, or recalibrate (Section 9). If recalibration is required, recalibration for the 209 Congeners (Section 9) must also be performed.

12.1.3 Retention Times (RTs) and Gas Chromatograph (GC) Resolution

12.1.3.1 Retention Times

12.1.3.1.1 The absolute RTs of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12) in the verification test (Section 12.1.2) must be within ±15 sec. of the respective RTs in the calibration or, if an alternate column or column system is employed, within ±15 sec. of the respective RTs in the calibration for the alternate column or column system (Section 6.8).

12.1.3.1.2 The Relative Retention Times (RRTs) of native CB congeners and labeled compounds in the verification test (Section 12.1.2) must be within their respective RRT limits in Table 2 or, if an alternate column or column system is employed, within their respective RRT limits for the alternate column or column system (Section 6.8).

12.1.3.1.3 If the absolute or RRT of any compound is not within the limits specified, the GC is not performing properly. In this event, adjust the GC and repeat the verification test (Section 12.1.2) or recalibrate (Section 9), or replace the GC column and verify either calibration or recalibrate.

12.1.3.2 GC Resolution

12.1.3.2.1 As a final step in calibration verification, inject the diluted combined 209-Congener Standard Solution (Section 7.10.2.2 and Table 5).

12.1.3.2.2 The resolution specifications in Sections 6.8 must be met for the SPB-octyl column or, if an alternate column or column system is employed, must be met as specified for the alternate column or column system (Section 6.9.1.2). If these specifications are not met, the GC analysis conditions must be adjusted until the specifications are met, or the column must be replaced and the calibration verification tests repeated (Section 12.1.2), or the system must be recalibrated (Section 9).

12.1.3.2.3 After the resolution specifications are met, update the RTs, RRTs, and RRFs for all of the congeners except the Toxics and LOC CB congeners. For the Toxics and LOC CB congeners, the multi-point calibration data must be used (Section 9).

12.2 Method Blank

12.2.1 Summary of Method Blanks

A method blank is a volume or weight of a clean reference matrix (reagent water for aqueous samples, sand for soil/sediment samples, or corn oil for tissue samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of the method blank is to determine the levels of contamination associated with the processing and analysis of the samples.

12.2.2 Frequency of Method Blanks

A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank will not exceed 20 field samples [excluding Performance Evaluation (PE) samples]. In addition, a method blank will:

- Be extracted by the same procedure used to extract samples.
- Be analyzed on each HRGC/HRMS system used to analyze associated samples.

12.2.3 Procedure for Method Blank Preparation

A method blank for aqueous samples consists of 1 L of reagent water spiked with 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12). For soil/sediment samples, a method blank consists of 10 g of sand spiked with 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12). For tissue samples, a method blank consists of 10 g of corn oil spiked with 1 mL of the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12).

12.2.4 Technical Acceptance Criteria for Method Blank Analysis

12.2.4.1 All blanks must be extracted and analyzed at the frequency described in Section 12.2.2 on an HRGC/HRMS system meeting all the technical acceptance criteria in Section 12.1.

12.2.4.2 The blank must meet the sample acceptance criteria listed in Section 11.0.

12.2.4.3 For the 12 Toxics, the method blank must contain less than the Contract Required Quantitation Limit (CRQL) of any single toxic congener (Exhibit C).

12.2.5 Corrective Action for Method Blanks

12.2.5.1 If a method blank does not meet the technical acceptance criteria for method blank analysis, the Contractor must consider the analytical system to be out of control.

12.2.5.2 If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures taken and documented before further sample analysis proceeds. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the HRGC/HRMS is eliminated. Samples associated with the contaminated blank must be reextracted and reanalyzed at no additional cost to USEPA.

12.3 Initial Demonstration of Laboratory Ability

Before the analysis of any field samples under this contract, the Contractor will demonstrate the ability to generate acceptable precision and recovery with this method and to meet the CRQL requirements. The Contractor will retain copies of the raw data and calculations from these studies and any additional studies carried out during the contract.

12.3.1 Initial Precision and Recovery (IPR)

12.3.1.1 For aqueous samples, extract, concentrate, and analyze four 1 L aliquots of reagent water spiked with 1 mL each of the Native Toxics/LOC spiking solution (Section 7.11), the Labeled Toxics/LOC/Window-Defining Congeners Standard Spiking Solution (Section 7.12), and the Labeled Cleanup Standard Spiking Solution (Section 7.13), according to the procedures in Section 10. For soil/sediment or tissue samples, four aliquots of the appropriate reference matrix (Section 7.6) are used. All sample processing steps that are to be used must be included in this test.

12.3.1.2 Using results of the set of four analyses, compute the average Percent Recovery (%R) of the extracts and the Relative Standard Deviation (RSD) of the concentration for each compound, by isotope dilution for CB congeners with a labeled analog, and by Internal Standard for CB congeners without a labeled analog and for the labeled compounds.

12.3.1.3 For each CB and labeled compound, compare the RSD and %R with the corresponding limits for initial precision and recovery in Table 6. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable. However, if any individual RSD exceeds the precision limit, or any individual %R falls outside the range for recovery, system performance is unacceptable for that compound. Correct the problem and repeat the test.

12.3.2 Method Detection Limits (MDL)

12.3.2.1 For each matrix and extraction/cleanup procedure, the Contractor will carry out an MDL study meeting the requirements in 40 CFR Part 136, Appendix B, for each Toxic congener. The MDLs for each Toxic congener determined by these studies will be less than the CRQL listed for that Toxic congener and matrix in Exhibit C.

13.0 METHOD PERFORMANCE

Not Applicable.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. When feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasiblely reduced at the source, USEPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applied to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society at http://membership.acs.org/c/ccs/pub_9.htm.

15.0 WASTE MANAGEMENT

USEPA requires that laboratory waste management practices be conducted consistently with all applicable rule and regulations for Federal, State, and Local governments. USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and by complying with all solid and hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel", available from the American Chemical Society available from the American Chemical Society's Office of Legislative and Government Affairs, 1155 16th Street NW, Washington, D.C. 20036, (202) 872-4386.

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17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table 1. Names, International Union of Pure and Applied Chemistry (IUPAC) Numbers, and Chemical Abstracts Service (CAS) Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS.

CB Congener[1]	IUPAC Number	CAS Registry Number	Labeled Analog	IUPAC Analog	CAS Registry Number
2-MoCB	1	2051-60-7	$^{13}C_{12}$-2-MoCB[2]	1L	234432-85-0
3-MoCB	2	2051-61-8			
4-MoCB	3	2051-62-9	$^{13}C_{12}$-4-MoCB[2]	3L	208263-77-8
2,2'-DiCB	4	13029-08-8	$^{13}C_{12}$-2,2'-DiCB[2]	4L	234432-86-1
2,3-DiCB	5	16605-91-7			
2,3'-DiCB	6	25569-80-6			
2,4-DiCB	7	33284-50-3			
2,4'-DiCB[3]	8	34883-43-7			
2,5-DiCB	9	34883-39-1	$^{13}C_{12}$-2,5-DiCB[4]	9L	250694-89-4
2,6-DiCB	10	33146-45-1			
3,3'-DiCB	11	2050-67-1			
3,4-DiCB	12	2974-92-7			
3,4'-DiCB	13	2974-90-5			
3,5-DiCB	14	34883-41-5			
4,4'-DiCB	15	2050-68-2	$^{13}C_{12}$-4,4'-DiCB[2]	15L	208263-67-6
2,2',3-TrCB	16	38444-78-9			
2,2',4-TrCB	17	37680-66-3			
2,2',5-TrCB[3]	18	37680-65-2			
2,2',6-TrCB	19	38444-73-4	$^{13}C_{12}$-2,2',6-TrCB[2]	19L	234432-87-2
2,3,3'-TrCB	20	38444-84-7			
2,3,4-TrCB	21	55702-46-0			
2,3,4'-TrCB	22	38444-85-8			
2,3,5-TrCB	23	55720-44-0			
2,3,6-TrCB	24	55702-45-9			
2,3',4-TrCB	25	55712-37-3			
2,3',5-TrCB	26	38444-81-4			
2,3',6-TrCB	27	38444-76-7			
2,4,4'-TrCB[3]	28	7012-37-5	$^{13}C_{12}$-2,4,4'-TriCB[5]	28L	208263-76-7
2,4,5-TrCB	29	15862-07-4			
2,4,6-TrCB	30	35693-92-6			
2,4',5-TrCB	31	16606-02-3			
2,4',6-TrCB	32	38444-77-8			
2',3,4-TrCB	33	38444-86-9			
2',3,5-TrCB	34	37680-68-5			
3,3',4-TrCB	35	37680-69-6			
3,3',5-TrCB	36	38444-87-0			
3,4,4'-TrCB	37	38444-90-5	$^{13}C_{12}$-3,4,4'-TrCB[2]	37L	208263-79-0
3,4,5-TrCB	38	53555-66-1			
3,4',5-TrCB	39	38444-88-1			
2,2',3,3'-TeCB	40	38444-93-8			
2,2',3,4-TeCB	41	52663-59-9			
2,2',3,4'-TeCB	42	36559-22-5			
2,2',3,5-TeCB	43	70362-46-8			
2,2',3,5'-TeCB[3]	44	41464-39-5			

CB Congener[1]	**IUPAC Number**	**CAS Registry Number**	**Labeled Analog**	**IUPAC Analog**	**CAS Registry Number**
2,2',3,6-TeCB	45	70362-45-7			
2,2',3,6'-TeCB	46	41464-47-5			
2,2',4,4'-TeCB	47	2437-79-8			
2,2',4,5-TeCB	48	70362-47-9			
2,2',4,5'-TeCB	49	41464-40-8			
2,2',4,6-TeCB	50	62796-65-0			
2,2',4,6'-TeCB	51	68194-04-7			
2,2',5,5'-TeCB[3]	52	35693-99-3	$^{13}C_{12}$-2,2',5,5'-TeCB[4]	52L	208263-80-3
2,2',5,6'-TeCB	53	41464-41-9			
2,2',6,6'-TeCB	54	15968-05-5	$^{13}C_{12}$-2,2',6,6'-TeCB[2]	54L	234432-88-3
2,3,3',4'-TeCB	55	74338-24-2			
2,3,3',4'-TeCB	56	41464-43-1			
2,3,3',5-TeCB	57	70424-67-8			
2,3,3',5'-TeCB	58	41464-49-7			
2,3,3',6-TeCB	59	74472-33-6			
2,3,4,4'-TeCB	60	33025-41-1			
2,3,4,5-TeCB	61	33284-53-6			
2,3,4,6-TeCB	62	54230-22-7			
2,3,4',5-TeCB	63	74472-34-7			
2,3,4',6-TeCB	64	52663-58-8			
2,3,5,6-TeCB	65	33284-54-7			
2,3',4,4'-TeCB[3]	66	32598-10-0			
2,3',4,5-TeCB	67	73575-53-8			
2,3',4,5'-TeCB	68	73575-52-7			
2,3',4,6-TeCB	69	60233-24-1			
2,3',4',5-TeCB	70	32598-11-1			
2,3',4',6-TeCB	71	41464-46-4			
2,3',5,5'-TeCB	72	41464-42-0			
2,3',5',6-TeCB	73	74338-23-1			
2,4,4',5-TeCB	74	32690-93-0			
2,4,4',6-TeCB	75	32598-12-2			
2',3,4,5-TeCB	76	70362-48-0			
3,3',4,4'-TeCB[3,6]	77	32598-13-3	$^{13}C_{12}$-3,3',4,4'-TeCB[2,7]	77L	105600-23-5
3,3',4,5-TeCB	78	70362-49-1			
3,3',4,5'-TeCB	79	41464-48-6			
3,3',5,5'-TeCB	80	33284-52-5			
3,4,4',5-TeCB[6]	81	70362-50-4	$^{13}C_{12}$-3,4,4',5-TeCB[7]	81L	208461-24-9
2,2',3,3',4-PeCB	82	52663-62-4			
2,2',3,3',5-PeCB	83	60145-20-2			
2,2',3,3',6-PeCB	84	52663-60-2			
2,2',3,4,4'-PeCB	85	65510-45-4			
2,2',3,4,5-PeCB	86	55312-69-1			
2,2',3,4,5'-PeCB	87	38380-02-8			
2,2',3,4,6-PeCB	88	55215-17-3			
2,2',3,4,6'-PeCB	89	73575-57-2			
2,2',3,4',5-PeCB	90	68194-07-0			
2,2',3,4',6-PeCB	91	68194-05-8			
2,2',3,5,5'-PeCB	92	52663-61-3			
2,2',3,5,6-PeCB	93	73575-56-1			
2,2',3,5,6'-PeCB	94	73575-55-0			

CB Congener[1]	IUPAC Number	CAS Registry Number	Labeled Analog	IUPAC Analog	CAS Registry Number
2,2',3,5',6-PeCB	95	38379-99-6			
2,2',3,6,6'-PeCB	96	73575-54-9			
2,2',3',4,5-PeCB	97	41464-51-1			
2,2',3',4,6-PeCB	98	60233-25-2			
2,2',4,4',5-PeCB	99	38380-01-7			
2,2',4,4',6-PeCB	100	39485-83-1			
2,2',4,5,5'-PeCB[3]	101	37680-73-2	$^{13}C_{12}$-2,2',4,5,5'-PeCB[4]	101L	104130-39-4
2,2',4,5,6'-PeCB	102	68194-06-9			
2,2',4,5,'6-PeCB	103	60145-21-3			
2,2',4,6,6'-PeCB	104	56558-16-8	$^{13}C_{12}$-2,2',4,6,6'-PeCB[2]	104L	234432-89-4
2,3,3',4,4'-PeCB[3,6]	105	32598-14-4	$^{13}C_{12}$-2,3,3',4,4'-PeCB[7]	105L	208263-62-1
2,3,3',4,5-PeCB	106	70424-69-0			
2,3,3',4',5-PeCB	107	70424-68-9			
2,3,3',4,5'-PeCB	108	70362-41-3			
2,3,3',4,6-PeCB	109	74472-35-8			
2,3,3',4',6-PeCB	110	38380-03-9			
2,3,3',5,5'-PeCB	111	39635-32-0	$^{13}C_{12}$-2,3,3',5,5'-PeCB[5]	111 L	235416-29-2
2,3,3',5,6-PeCB	112	74472-36-9			
2,3,3',5',6-PeCB	113	68194-10-5			
2,3,4,4',5-PeCB[6]	114	74472-37-0	$^{13}C_{12}$-2,3,4,4',5-PeCB[7]	114 L	208263-63-2
2,3,4,4',6-PeCB	115	74472-38-1			
2,3,4,5,6-PeCB	116	18259-05-7			
2,3,4',5,6-PeCB	117	68194-11-6			
2,3',4,4',5-PeCB[3,6]	118	31508-00-6	$^{13}C_{12}$-2,3',4,4',5-PeCB[7]	118 L	104130-40-7
2,3',4,4',6-PeCB	119	56558-17-9			
2,3',4,5,5'-PeCB	120	68194-12-7			
2,3',4,5,'6-PeCB	121	56558-18-0			
2',3,3',4,5-PeCB	122	76842-07-4			
2',3,4,4',5-PeCB[6]	123	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-PeCB[7]	123L	208263-64-3
2',3,4,5,5'-PeCB	124	70424-70-3			
2',3,4,5,6'-PeCB	125	74472-39-2			
3,3',4,4',5-PeCB[3,6]	126	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-PeCB[2,7]	126L	208263-65-4
3,3',4,5,5'-PeCB	127	39635-33-1			
2,2',3,3',4,4'-HxCB[3]	128	38380-07-3			
2,2',3,3',4,5-HxCB	129	55215-18-4			
2,2',3,3',4,5'-HxCB	130	52663-66-8			
2,2',3,3',4,6-HxCB	131	61798-70-7			
2,2',3,3',4,6'-HxCB	132	38380-05-1			
2,2',3,3',5,5'-HxCB	133	35694-04-3			
2,2',3,3',5,6-HxCB	134	52704-70-8			
2,2',3,3',5,6'-HxCB	135	52744-13-5			
2,2',3,3',6,6'-HxCB	136	38411-22-2			
2,2',3,4,4',5-HxCB	137	35694-06-5			
2,2',3,4,4',5'-HxCB[3]	138	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[4]	138L	208263-66-5
2,2',3,4,4',6-HxCB	139	56030-56-9			
2,2',3,4,4',6'-HxCB	140	59291-64-4			
2,2',3,4,5,5'-HxCB	141	52712-04-6			
2,2',3,4,5,6-HxCB	142	41411-61-4			
2,2',3,4,5,6'-HxCB	143	68194-15-0			
2,2',3,4,5',6-HxCB	144	68194-14-9			

CB Congener[1]	IUPAC Number	CAS Registry Number	Labeled Analog	IUPAC Analog	CAS Registry Number
2,2',3,4,6,6'-HxCB	145	74472-40-5			
2,2',3,4',5,5'-HxCB	146	51908-16-8			
2,2',3,4',5,6-HxCB	147	68194-13-8			
2,2',3,4',5,6'-HxCB	148	74472-41-6			
2,2',3,4',5',6-HxCB	149	38380-04-0			
2,2',3,4',6,6'-HxCB	150	68194-08-1			
2,2',3,5,5',6-HxCB	151	52663-63-5			
2,2',3,5,6,6'-HxCB	152	68194-09-2			
2,2',4,4',5,5'-HxCB[3]	153	35065-27-1			
2,2',4,4',5',6-HxCB	154	60145-22-4			
2,2',4,4',6,6'-HxCB	155	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-HxCB[2]	155L	234432-90-7
2,3,3',4,4',5-HxCB[6]	156	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-HxCB[7]	156L	208263-68-7
2,3,3',4,4',5'-HxCB[6]	157	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[7]	157L	235416-30-5
2,3,3',4,4',6-HxCB	158	74472-42-7			
2,3,3',4,5,5'-HxCB	159	39635-35-3			
2,3,3',4,5,6-HxCB	160	41411-62-5			
2,3,3',4,5',6-HxCB	161	74472-43-8			
2,3,3',4',5,5'-HxCB	162	39635-34-2			
2,3,3',4',5,6-HxCB	163	74472-44-9			
2,3,3',4',5',6-HxCB	164	74472-45-0			
2,3,3',5,5',6-HxCB	165	74472-46-1			
2,3,4,4',5,6-HxCB	166	41411-63-6			
2,3',4,4',5,5'-HxCB[6]	167	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[7]	167L	208263-69-8
2,3',4,4',5',6-HxCB	168	59291-65-5			
3,3',4,4',5,5'-HxCB[3,6]	169	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[2,7]	169L	208263-70-1
2,2',3,3',4,4',5-HpCB[3]	170	35065-30-6			
2,2'3,3',4,4',6-HpCB	171	52663-71-5			
2,2',3,3',4,5,5'-HpCB	172	52663-74-8			
2,2',3,3',4,5,6-HpCB	173	68194-16-1			
2,2',3,3',4,5,6'-HpCB	174	38411-25-5			
2,2',3,3',4,5',6-HpCB	175	40186-70-7			
2,2',3,3',4,6,6'-HpCB	176	52663-65-7			
2,2',3,3',4',5,6-HpCB	177	52663-70-4			
2,2',3,3',5,5',6-HpCB	178	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[5]	178L	232919-67-4
2,2',3,3',5,6,6'-HpCB	179	52663-64-6			
2,2',3,4,4',5,5'-HpCB[3]	180	35065-29-3			
2,2',3,4,4',5,6-HpCB	181	74472-47-2			
2,2',3,4,4',5,6'-HpCB	182	60145-23-5			
2,2',3,4,4',5',6-HpCB	183	52663-69-1			
2,2',3,4,4',6,6'-HpCB	184	74472-48-3			
2,2',3,4,5,5',6-HpCB	185	52712-05-7			
2,2',3,4,5,6,6'-HpCB	186	74472-49-4			
2,2',3,4',5,5',6-HpCB[3]	187	52663-68-0			
2,2',3,4',5,6,6'-HpCB	188	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[2]	188L	234432-91-8
2,3,3',4,4',5,5'-HpCB[6]	189	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB[2,7]	189L	208263-73-4
2,3,3',4,4',5,6-HpCB	190	41411-64-7			
2,3,3',4,4',5',6-HpCB	191	74472-50-7			
2,3,3',4,5,5',6-HpCB	192	74472-51-8			
2,3,3',4',5,5',6-HpCB	193	69782-91-8			
2,2',3,3',4,4',5,5'-OcCB	194	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB[4]	194L	208263-74-5
2,2',3,3',4,4',5,6-OcCB[3]	195	52663-78-2			

CB Congener[1]	IUPAC Number	CAS Registry Number	Labeled Analog	IUPAC Analog	CAS Registry Number
2,2',3,3',4,4',5,6'-OcCB	196	42740-50-1			
2,2',3,3',4,4',6,6'-OcCB	197	33091-17-7			
2,2',3,3',4,5,5',6-OcCB	198	68194-17-2			
2,2',3,3',4,5,5',6'-OcCB	199	52663-75-9			
2,2',3,3',4,5,6,6'-OcCB	200	52663-73-7			
2,2',3,3',4,5',6,6'-OcCB	201	40186-71-8			
2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[2]	202L	105600-26-8
2,2',3,4,4',5,5',6-OcCB	203	52663-76-0			
2,2',3,4,4',5,6,6'-OcCB	204	74472-52-9			
2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[2]	205L	234446-64-1
2,2',3,3',4,4',5,5',6-NoCB[3]	206	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB[2]	206L	208263-75-6
2,2',3,3',4,4',5,6,6'-NoCB	207	52663-79-3			
2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[2]	208L	234432-92-9
DeCB[3]	209	2051-24-3	$^{13}C_{12}$-DeCB[2]	209L	105600-27-9

Table 2. Retention Times (RTs), RT References, Relative Retention Times (RRTs), Estimated Method Detection Limits (EMDLs), and Estimated Quantitation Limits for the 209 CB Congeners on SPB-Octyl.

Cl No.[8]	**IUPAC No.** [9,10]	**RT Ref**[11]	**RTs**[12]	**RRT**[13]	**RRT Limits**[14]	**Window (sec)**[15]	**Quantitation Reference**[16]	**Estimated Detection and Quantitation Limits - Matrix and Concentration**[17]				
								Water (pg/L)		**Other (ng/kg)**		**Extract (pg/µL)**
								EMDL	**EML**	**EMDL**	**EML**	**EML**
Compounds using 9L ($^{13}C_{12}$-2,5-DiCB) as Labeled Internal Standard												
CB Congener												
Monochlorobiphenyls												
1	1	1L	13:44	1.0012	0.9951-1.0073	10	1L	82	200	8	20	10
1	2	3L	16:08	0.9878	0.9847-0.9908	6	1L/3L	4	10	0.4	1	0.5
1	3	3L	16:21	1.0010	0.9980-1.0041	6	3L	88	200	9	20	10
Dichlorobiphenyls												
2	4	4L	16:40	1.0010	0.9960-1.0060	10	4L	172	500	17	50	20
2	10	4L	16:53	1.0140	1.0110-1.0170	6	4L/15L	22	50	2	5	2
2	9	4L	18:55	1.1361	1.1331-1.1391	6	4L/15L	20	50	2	5	2
2	7	4L	19:07	1.1481	1.1451-1.1512	6	4L/15L	15	50	2	5	2
2	6	4L	19:26	1.1672	1.1642-1.1702	6	4L/15L	13	50	1	5	2
2	5	4L	19:48	1.1892	1.1862-1.1922	6	4L/15L	11	50	1	5	2
2	8	4L	19:56	1.1972	1.1942-1.2002	6	4L/15L	121	500	12	50	20
2	14	15L	21:42	0.9267	0.9246-0.9288	6	4L/15L	31	100	3	10	5
2	11	15L	22:42	0.9694	0.9673-0.9715	6	4L/15L	105	200	10	20	10
2	13	15L	23:03	0.9843	0.9822-0.9865	6	4L/15L					
2	12	15L	23:06	0.9865	0.9843-0.9886	6	4L/15L	28	100	3	10	5
2	13/12	15L	23:04	0.9851	0.9829-0.9872	6	4L/15L					
2	15	15L	23:26	1.0007	0.9972-1.0043	10	15L	183	500	18	50	20

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
	Trichlorobiphenyls											
3	19	19L	20:19	1.0008	0.9967-1.0049	10	19L	42	100	4	10	5
3	30	19L	22:15	1.0961	1.0936-1.0985	6	19L/37L	175	500	17	50	20
3	18	19L	22:23	1.1026	1.1002-1.1051	6	19L/37L					
3	30/18	19L	22:19	1.0993	1.0969-1.1018	6	19L/37L					
3	17	19L	22:49	1.1240	1.1215-1.1264	6	19L/37L	86	200	9	20	10
3	27	19L	23:06	1.1379	1.1355-1.1404	6	19L/37L	59	200	6	20	10
3	24	19L	23:14	1.1445	1.1420-1.1470	6	19L/37L	53	200	5	20	10
3	16	19L	23:25	1.1535	1.1511-1.1560	6	19L/37L	35	100	4	10	5
3	32	19L	24:57	1.2291	1.2266-1.2315	6	19L/37L	84	200	8	20	10
3	34	19L	25:17	1.2455	1.2430-1.2479	6	19L/37L	74	200	7	20	10
3	23	19L	25:26	1.2529	1.2504-1.2553	6	19L/37L	50	200	5	20	10
3	29	19L	25:47	1.2701	1.2660-1.2742	10	19L/37L	83	200	8	20	10
3	26	19L	25:48	1.2709	1.2668-1.2750	10	19L/37L					
3	26/29	19L	25:48	1.2709	1.2668-1.2750	10	19L/37L					
3	25	37L	26:04	0.8364	0.8348-0.8380	6	19L/37L	55	200	5	20	10
3	31	37L	26:25	0.8476	0.8460-0.8492	6	19L/37L	152	500	15	50	20
3	28	37L	26:44	0.8578	0.8551-0.8604	10	19L/37L	192	500	19	50	20
3	20	37L	26:49	0.8604	0.8578-0.8631	10	19L/37L					
3	28/20	37L	26:47	0.8594	0.8567-0.8620	10	19L/37L					
3	21	37L	26:58	0.8652	0.8626-0.8679	10	19L/37L	51	200	5	20	10
3	33	37L	27:01	0.8668	0.8642-0.8695	10	19L/37L					
3	21/33	37L	26:59	0.8658	0.8631-0.8684	10	19L/37L					

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
3	22	37L	27:29	0.8818	0.8802-0.8834	6	19L/37L	90	200	9	20	10
3	36	37L	29:05	0.9332	0.9316-0.9348	6	19L/37L	79	200	8	20	10
3	39	37L	29:30	0.9465	0.9449-0.9481	6	19L/37L	85	200	9	20	10
3	38	37L	30:10	0.9679	0.9663-0.9695	6	19L/37L	83	200	8	20	10
3	35	37L	30:42	0.9850	0.9834-0.9866	6	19L/37L	77	200	8	20	10
3	37	37L	31:11	1.0005	0.9989-1.0021	6	37L	132	500	13	50	20
Labeled Compounds												
1	1L	9L	13:43	0.7257	0.7125-0.7390	30	9L					
1	3L	9L	16:20	0.8642	0.8510-0.8774	30	9L					
2	4L	9L	16:39	0.8810	0.8677-	30	9L					
2	15L	9L	23:25	1.2390	1.2302-	20	9L					
3	19L	9L	20:118	1.0741	1.0608-1.0873	30	9L					
3	37L	52L	31:10	1.0803	1.0716-1.0890	30	52L					
Compounds using 52L ($^{13}C_{12}$-2,2',5,5'-TeCB) as Labeled Internal Standard												
CB Congener												
Tetrachlorobiphenyls												
4	54	54L	23:51	1.0007	0.9972-1.0042	10	54L	118	500	12	50	20
4	50	54L	26:07	1.0958	1.0923-1.0993	10	54L/81L/77L	58	200	6	20	10
4	53	54L	26:09	1.0972	1.0937-1.1007	10	54L/81L/77L					
4	50/53	54L	26:08	1.0965	1.0930-1.1000	10	54L/81L/77L					
4	45	54L	26:55	1.1294	1.1259-1.1329	10	54L/81L/77L	51	200	5	20	10
4	51	54L	26:58	1.1315	1.1280-1.1350	10	54L/81L/77L					
4	45/51	54L	26:57	1.1308	1.1273-1.1343	10	54L/81L/77L					
4	46	54L	27:18	1.1455	1.1434-1.1476	6	54L/81L/77L	101	200	10	20	10

Cl No.[8]	IUPAC No.[9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
4	52	54L	28:45	1.2063	1.2042-1.2084	6	54L/81L/77L	191	500	19	50	20
4	73	54L	28:52	1.2112	1.2091-1.2133	6	54L/81L/77L	160	500	16	50	20
4	43	54L	28:58	1.2154	1.2133-1.2175	6	54L/81L/77L	94	200	9	20	10
4	69	54L	29:08	1.2224	1.2189-1.2259	10	54L/81L/77L	115	500	11	50	20
4	49	54L	29:16	1.2280	1.2245-1.2315	10	54L/81L/77L					
4	69/49	54L	29:12	1.2252	1.2217-1.2287	10	54L/81L/77L					
4	48	54L	29:33	1.2399	1.2378-1.2420	6	54L/81L/77L	76	200	8	20	10
4	65	54L	29:49	1.2510	1.2476-1.2545	10	54L/81L/77L	195	500	19	50	20
4	47	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L					
4	44	54L	29:53	1.2538	1.2503-1.2573	10	54L/81L/77L					
4	44/47/65	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L					
4	62	54L	30:06	1.2629	1.2594-1.2664	10	54L/81L/77L	57	200	6	20	10
4	75	54L	30:08	1.2643	1.2608-1.2678	10	54L/81L/77L					
4	59	54L	30:12	1.2671	1.2636-1.2706	10	54L/81L/77L					
4	59/62/75	54L	30:09	1.2650	1.2615-1.2685	10	54L/81L/77L					
4	42	54L	30:26	1.2769	1.2748-1.2790	6	54L/81L/77L	61	200	6	20	10
4	41	54L	30:52	1.2951	1.2916-1.2986	10	54L/81L/77L	119	500	12	50	20
4	71	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L					
4	40	54L	30:01	1.2594	1.2559-1.2629	10	54L/81L/77L					
4	41/40/71	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L					
4	64	54L	31:12	1.3091	1.3070-1.3112	6	54L/81L/77L	70	200	7	20	10
4	72	81L	31:59	0.8336	0.8323-0.8349	6	54L/81L/77L	158	500	16	50	20

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	**EML**	**EMDL**	**EML**	**EML**
4	68	81L	32:18	0.8419	0.8406-0.8432	6	54L/81L/77L	149	500	15	50	20
4	57	81L	32:46	0.8540	0.8527-0.8553	6	54L/81L/77L	125	500	12	50	20
4	58	81L	33:05	0.8623	0.8610-0.8636	6	54L/81L/77L	127	500	13	50	20
4	67	81L	33:13	0.8658	0.8645-0.8671	6	54L/81L/77L	147	500	15	50	20
4	63	81L	33:30	0.8732	0.8719-0.8745	6	54L/81L/77L	138	500	14	50	20
4	61	81L	33:46	0.8801	0.8775-0.8827	12	54L/81L/77L	171	500	17	50	20
4	70	81L	33:53	0.8831	0.8805-0.8858	12	54L/81L/77L					
4	76	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L					
4	74	54L	33:57	0.8849	0.8827-0.8871	10	54L/81L/77L					
4	61/70/74/76	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L					
4	66	81L	34:15	0.8927	0.8914-0.8940	6	54L/81L/77L	162	500	16	50	20
4	55	81L	34:28	0.8983	0.8970-0.8997	6	54L/81L/77L	120	500	12	50	20
4	56	81L	35:03	0.9136	0.9123-0.9149	6	54L/81L/77L	98	200	10	20	10
4	60	81L	35:16	0.9192	0.9179-0.9205	6	54L/81L/77L	131	500	13	50	20
4	80	81L	35:32	0.9262	0.9248-0.9275	6	54L/81L/77L	175	500	18	50	20
4	79	81L	37:16	0.9713	0.9700-0.9726	6	54L/81L/77L	173	500	17	50	20
4	78	81L	37:52	0.9870	0.9857-0.9883	6	54L/81L/77L	171	500	17	50	20
4	81	81L	38:23	1.0004	0.9991-1.0017	6	81L	177	500	18	50	20
4	77	77L	39:02	1.0004	0.9991-1.0017	6	77L	169	500	17	50	20
	Labeled Compounds											
4	54L	52L	23:50	0.8261	0.8203-0.8319	20	52L					
4	81L	52L	38:22	1.3299	1.3241-1.3356	20	52L					
4	77L	52L	39:01	1.3524	1.3466-1.3582	20	52L					

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
Compounds using 101L ($^{13}C_{12}$-2,2',4,5,5'-PeCB) as Labeled Internal Standard												
CB Congener												
Pentachlorobiphenyls												
5	104	104L	29:46	1.0000	0.9972-1.0028	10	104L	228	500	23	50	20
5	96	104L	30:17	1.0174	1.0146-1.0202	10	104L/123L/114L/118L/105L/126L	210	500	21	50	20
5	103	104L	32:11	1.0812	1.0795-1.0829	6	104L/123L/114L/118L/105L/126L	225	500	23	50	20
5	94	104L	32:29	1.0913	1.0896-1.0929	6	104L/123L/114L/118L/105L/126L	121	500	12	50	20
5	95	104L	33:00	1.1086	1.1058-1.1114	10	104L/123L/114L/118L/105L/126L	221	500	22	50	20
5	100	104L	33:06	1.1120	1.1092-1.1148	10	104L/123L/114L/118L/105L/126L					
5	93	104L	33:14	1.1165	1.1137-1.1193	10	104L/123L/114L/118L/105L/126L					
5	102	104L	33:21	1.1204	1.1176-1.1232	10	104L/123L/114L/118L/105L/126L					
5	98	104L	33:26	1.1232	1.1204-1.1260	10	104L/123L/114L/118L/105L/126L					
5	95/100/93/102/98	104L	33:13	1.1159	1.1131-1.1187	15	104L/123L/114L/118L/105L/126L					
5	88	104L	33:48	1.1355	1.1321-1.1389	12	104L/123L/114L/118L/105L/126L	118	500	12	50	20
5	91	104L	33:55	1.1394	1.1366-1.1422	10	104L/123L/114L/118L/105L/126L					
5	88/91	104L	33:52	1.1377	1.1344-1.1411	12	104L/123L/114L/118L/105L/126L					
5	84	104L	34:14	1.1501	1.1484-1.1517	6	104L/123L/114L/118L/105L/126L	124	500	12	50	20
5	89	104L	34:44	1.1669	1.1652-1.1685	6	104L/123L/114L/118L/105L/126L	195	500	19	50	20
5	121	104L	34:57	1.1741	1.1725-1.1758	6	104L/123L/114L/118L/105L/126L	209	500	21	50	20
5	92	123L	35:26	0.8639	0.8627-0.8651	6	104L/123L/114L/118L/105L/126L	115	500	12	50	20
5	113	104L	36:01	0.8781	0.8761-0.8801	10	104L/123L/114L/118L/105L/126L	241	1000	24	100	50
5	90	104L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L/105L/126L					
5	101	104L	36:04	0.8793	0.8773-0.8813	10	104L/123L/114L/118L/105L/126L					

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17] Water (pg/L) EMDL	Water (pg/L) EML	Other (ng/kg) EMDL	Other (ng/kg) EML	Extract (pg/µL) EML
5	113/90/101	104L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L/105L/126L					
5	83	104L	36:39	0.8935	0.8911-0.8960	12	104L/123L/114L/118L/105L/126L	217	500	22	50	20
5	99	104L	36:41	0.8944	0.8923-0.8964	10	104L/123L/114L/118L/105L/126L					
5	83/99	104L	36:40	0.8939	0.8915-0.8964	12	104L/123L/114L/118L/105L/126L					
5	112	104L	36:51	0.8984	0.8972-0.8996	6	104L/123L/114L/118L/105L/126L	245	1000	25	100	50
5	119	104L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L/105L/126L	149	500	15	50	20
5	108	104L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L/105L/126L					
5	86	104L	37:17	0.9090	0.9057-0.9122	16	104L/123L/114L/118L/105L/126L					
5	97	104L	37:17	0.9090	0.9057-0.9122	16	104L/123L/114L/118L/105L/126L					
5	125	104L	37:21	0.9106	0.9074-0.9139	16	104L/123L/114L/118L/105L/126L					
5	87	104L	37:25	0.9122	0.9102-0.9143	10	104L/123L/114L/118L/105L/126L					
5	108/119/86/97/125/87	104L	37:19	0.9098	0.9065-0.9130	16	104L/123L/114L/118L/105L/126L					
5	117	104L	37:57	0.9252	0.9228-0.9277	12	104L/123L/114L/118L/105L/126L	104	200	10	20	10
5	116	104L	38:02	0.9273	0.9248-0.9297	12	104L/123L/114L/118L/105L/126L					
5	85	104L	38:05	0.9285	0.9265-0.9305	10	104L/123L/114L/118L/105L/126L					
5	117/116/85	104L	38:00	0.9265	0.9240-0.9289	12	104L/123L/114L/118L/105L/126L					
5	110	104L	38:16	0.9330	0.9309-0.9350	10	104L/123L/114L/118L/105L/126L	243	1000	24	100	50
5	115	104L	38:18	0.9338	0.9317-0.9358	10	104L/123L/114L/118L/105L/126L					
5	110/115	104L	38:17	0.9334	0.9313-0.9354	10	104L/123L/114L/118L/105L/126L					
5	82	104L	38:40	0.9427	0.9415-0.9439	6	104L/123L/114L/118L/105L/126L	133	500	13	50	20
5	111	104L	38:52	0.9476	0.9464-0.9488	6	104L/123L/114L/118L/105L/126L	243	1000	24	100	50
5	120	104L	39:21	0.9594	0.9581-0.9606	6	104L/123L/114L/118L/105L/126L	147	500	15	50	20

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
5	107	104L	40:39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L/105L/126L	200	1000	27	100	50
5	124	104L	40:40	0.9915	0.9894-0.9935	10	104L/123L/114L/118L/105L/126L					
5	107/124	104L	40:39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L/105L/126L					
5	109	104L	40:54	0.9972	0.9959-0.9984	6	104L/123L/114L/118L/105L/126L	103	200	10	20	10
5	123	123L	41:02	1.0004	0.9992-1.0016	6	123L	150	500	15	50	20
5	106	123L	41:10	1.0037	1.0024-1.0049	6	104L/123L/114L/118L/105L/126L	143	500	14	50	20
5	118	118L	41:22	1.0004	0.9992-1.0016	6	118L	193	500	19	50	20
5	122	118L	41:49	1.0113	1.0101-1.0125	6	104L/123L/114L/118L/105L/126L	117	500	12	50	20
5	114	114L	41:58	1.0004	0.9992-1.0016	6	114L	120	500	12	50	20
5	105	105L	42:43	0.9996	0.9984-1.0008	6	105L	109	200	11	20	10
5	127	105L	44:09	1.0332	1.0320-1.0343	6	104L/123L/114L/118L/105L/126L	278	1000	28	100	50
5	126	126L	45:58	1.0004	0.9993-1.0015	6	126L	136	500	14	50	20
Labeled Compounds												
5	104L	101L	29:46	0.8257	0.8211-0.8303	20	101L					
5	123L	101L	41:01	1.1378	1.1331-1.1424	20	101L					
5	118L	101L	41:21	1.1470	1.1424-1.1516	20	101L					
5	114L	101L	41:57	1.1637	1.1590-1.1683	20	101L					
5	105L	101L	42:44	1.1854	1.1808-1.1900	20	101L					
5	126L	101L	45:57	1.2746	1.2700-1.2792	20	101L					
Compounds using 138L ($^{13}C_{12}$-2,2',3,4,4',5'-HxCB) as Labeled Internal Standard												
CB Congener												
Hexachlorobiphenyls												
6	155	155L	35:44	1.0000	0.9977-1.0023	10	155L	339	1000	34	100	50
6	152	155L	36:07	1.0107	1.0093-1.0121	6	155L/156L/157L/167L	238	1000	24	100	50

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
6	150	155L	36:15	1.0145	1.0131-1.0159	6	155L/156L/157L/167L	328	1000	33	100	50
6	136	155L	36:44	1.0280	1.0266-1.0294	6	155L/156L/157L/167L	91	200	9	20	10
6	145	155L	37:00	1.0354	1.0340-1.0368	6	155L/156L/157L/167L	317	1000	32	100	50
6	148	155L	34:26	1.0756	1.0742-1.0770	6	155L/156L/157L/167L	324	1000	32	100	50
6	151	155L	39:10	1.0961	1.0938-1.0984	10	155L/156L/157L/167L	112	500	11	50	20
6	135	155L	39:17	1.0993	1.0970-1.1017	10	155L/156L/157L/167L					
6	154	155L	39:21	1.1012	1.0989-1.1035	10	155L/156L/157L/167L					
6	151/135/154	155L	39:15	1.0984	1.0961-1.1007	10	155L/156L/157L/167L					
6	144	155L	39:47	1.1133	1.1119-1.1147	6	155L/156L/157L/167L	167	500	17	50	20
6	147	155L	40:09	1.1236	1.1213-1.1259	10	155L/156L/157L/167L	179	500	18	50	20
6	149	155L	40:12	1.1250	1.1227-1.1273	10	155L/156L/157L/167L					
6	147/149	155L	40:10	1.1241	1.1217-1.1264	10	155L/156L/157L/167L					
6	134	155L	40:27	1.1320	1.1297-1.1343	10	155L/156L/157L/167L	134	500	13	50	20
6	143	155L	40:30	1.1334	1.1311-1.1357	10	155L/156L/157L/167L					
6	134/143	155L	40:29	1.1329	1.1306-1.1353	10	155L/156L/157L/167L					
6	139	155L	40:47	1.1413	1.1390-1.1437	10	155L/156L/157L/167L	196	500	20	50	20
6	140	155L	40:48	1.1418	1.1395-1.1441	10	155L/156L/157L/167L					
6	139/140	155L	40:47	1.1413	1.1390-1.1437	10	155L/156L/157L/167L					
6	131	155L	41:03	1.1488	1.1474-1.1502	6	155L/156L/157L/167L	121	500	12	50	20
6	142	155L	41:13	1.1535	1.1521-1.1549	6	155L/156L/157L/167L	311	1000	31	100	50
6	132	155L	41:36	1.1642	1.1618-1.1665	10	155L/156L/157L/167L	125	500	12	50	20
6	133	155L	41:57	1.1740	1.1726-1.1754	6	155L/156L/157L/167L	169	500	17	50	20

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
6	165	167L	42:23	0.8864	0.8853-0.8874	6	155L/156L/157L/167L	361	1000	36	100	50
6	146	167L	42:38	0.8916	0.8906-0.8926	6	155L/156L/157L/167L	182	500	18	50	20
6	161	167L	42:47	0.8947	0.8937-0.8958	6	155L/156L/157L/167L	352	1000	35	100	50
6	153	167L	43:17	0.9052	0.9035-0.9069	10	155L/156L/157L/167L	130	500	13	50	20
6	168	167L	43:21	0.9066	0.9048-0.9083	10	155L/156L/157L/167L					
6	153/168	167L	43:19	0.9059	0.9041-0.9076	10	155L/156L/157L/167L					
6	141	167L	43:34	0.9111	0.9101-0.9122	6	155L/156L/157L/167L	93	200	9	20	10
6	130	167L	44:01	0.9205	0.9195-0.9216	6	155L/156L/157L/167L	136	500	14	50	20
6	137	167L	44:14	0.9251	0.9240-0.9261	6	155L/156L/157L/167L	300	1000	30	100	50
6	164	167L	44:22	0.9278	0.9268-0.9289	6	155L/156L/157L/167L	136	500	14	50	20
6	138	167L	44:42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L	211	500	21	50	20
6	163	167L	44:42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L					
6	129	167L	44:47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L					
6	160	167L	44:53	0.9387	0.9369-0.9404	10	155L/156L/157L/167L					
6	138/163/129/160	167L	44:47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L					
6	158	167L	45:05	0.9428	0.9418-0.9439	6	155L/156L/157L/167L	96	200	10	20	10
6	166	167L	45:59	0.9617	0.9599-0.9634	10	155L/156L/157L/167L	124	500	12	50	20
6	128	167L	46:46	0.9651	0.9634-0.9669	10	155L/156L/157L/167L					
6	128/166	167L	46:04	0.9634	0.9617-0.9651	10	155L/156L/157L/167L					
6	159	167L	46:59	0.9826	0.9815-0.9836	6	155L/156L/157L/167L	348	1000	35	100	50
6	162	167L	47:18	0.9892	0.9881-0.9902	6	155L/156L/157L/167L	355	1000	35	100	50
6	167	167L	47:49	1.0000	0.9990-1.0010	6	155L/156L/157L/167L	115	500	11	50	20

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
6	156	156L/157L	49:05	0.9993	0.9983-1.0003	6	156L/157L	132	500	13	50	20
6	157	156L/157L	49:09	1.0007	0.9990-1.0024	10	156L/157L					
6	156/157	156L/157L	45:07	1.0000	0.9990-1.0010	6	156L/157L					
6	169	169L	52:31	0.9949	0.9940-0.9959	6	169L	161	500	16	50	20
Labeled Compounds												
6	155L	138L	35:44	0.7997	0.7960-0.8034	20	138L					
6	167L	138L	47:49	1.0701	1.0664-1.0739	20	138L					
6	156L	138L	49:05	1.0985	1.0974-1.0996	6	138L					
6	157L	138L	49:08	1.0996	1.0959-1.1033	20	138L					
6	156L/157L	138L	49:07	1.0992	1.0981-1.1003	6	138L					
6	169L	138L	52:30	1.1749	1.1738-1.1761	6	138L					
Compounds using 194L($^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB) as Labeled Internal Standard												
CB Congener												
Heptachlorobiphenyls												
7	188	188L	41:51	1.0000	0.9988-1.0012	6	188L	235	500	23	50	20
7	179	188L	42:19	1.0112	1.0100-1.0123	6	188L/189L	229	500	23	50	20
7	184	188L	42:45	1.0215	1.0203-1.0227	6	188L/189L	403	1000	40	100	50
7	176	188L	43:15	1.0335	1.0323-1.0346	6	188L/189L	385	1000	39	100	50
7	186	188L	43:45	1.0454	1.0442-1.0466	6	188L/189L	407	1000	41	100	50
7	178	188L	45:06	1.0777	1.0765-1.0789	6	188L/189L	221	500	22	50	20
7	175	188L	45:46	1.0936	1.0924-1.0948	6	188L/189L	383	1000	38	100	50
7	187	188L	46:02	1.1000	1.0988-1.1012	6	188L/189L	191	500	19	50	20
7	182	188L	46:14	1.1047	1.1035-1.1059	6	188L/189L	398	1000	40	100	50
7	183	188L	46:42	1.1159	1.1147-1.1171	6	188L/189L	401	1000	40	100	50

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
7	185	188L	46:53	1.1203	1.1191-1.1215	6	188L/189L					
7	183/185	188L	46:47	1.1179	1.1167-1.1191	6	188L/189L					
7	174	188L	47:02	1.1239	1.1227-1.1251	6	188L/189L	186	500	19	50	20
7	177	188L	47:30	1.1350	1.1338-1.1362	6	188L/189L	141	500	14	50	20
7	181	188L	47:52	1.1438	1.1426-1.1450	6	188L/189L	396	1000	40	100	50
7	171	188L	48:10	1.1509	1.1489-1.1529	10	188L/189L					
7	173	188L	48:11	1.1513	1.1501-1.1525	6	188L/189L	374	1000	37	100	50
7	171/173	188L	48:10	1.1509	1.1489-1.1529	10	188L/189L					
7	172	189L	49:47	0.9035	0.9026-0.9044	6	188L/189L	377	1000	38	100	50
7	192	189L	50:06	0.9093	0.9083-0.9102	6	188L/189L	420	1000	42	100	50
7	193	189L	50:26	0.9153	0.9144-0.9162	6	188L/189L					
7	180	189L	50:27	0.9156	0.9147-0.9165	6	188L/189L	136	500	14	50	20
7	180/193	189L	50:26	0.9153	0.9144-0.9162	6	188L/189L					
7	191	189L	50:51	0.9229	0.9220-0.9238	6	188L/189L	418	1000	42	100	50
7	170	189L	51:54	0.9419	0.9410-0.9428	6	188L/189L	162	500	16	50	20
7	190	189L	52:26	0.9516	0.9507-0.9525	6	188L/189L	234	500	23	50	20
7	189	189L	55:07	1.0003	0.9994-1.0012	6	189L	177	500	18	50	20
Octachlorobiphenyls												
8	202	202L	47:32	1.0004	0.9986-1.0021	10	202L	442	1000	44	100	50
8	201	202L	48:31	1.0210	1.0193-1.0228	10	202L/205L	440	1000	44	100	50
8	204	202L	49:11	1.0351	1.0340-1.0361	6	202L/205L	447	1000	45	100	50

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
8	197	202L	49:27	1.0407	1.0396-1.0417	6	202L/205L	245	1000	25	100	50
8	200	202L	49:40	1.0452	1.0442-1.0463	6	202L/205L					
8	197/200	202L	49:33	1.0428	1.0417-1.0438	6	202L/205L					
8	198	202L	52:30	1.1049	1.1031-1.1066	10	202L/205L	203	500	20	50	25
8	199	202L	52:32	1.1056	1.1045-1.1066	6	202L/205L					
8	198/199	202L	52:31	1.1052	1.1035-1.1070	10	202L/205L					
8	196	205L	53:13	0.9207	0.9198-0.9216	6	202L/205L	429	1000	43	100	50
8	203	205L	53:26	0.9245	0.9236-0.9253	6	202L/205L	444	1000	44	100	50
8	195	205L	54:55	0.9501	0.9493-0.9510	6	202L/205L	427	1000	43	100	50
8	194	205L	57:19	0.9916	0.9908-0.9925	6	202L/205L	170	500	17	50	20
8	205	205L	57:49	1.0003	0.9994-1.0012	6	205L	449	1000	45	100	50
Nonachlorobiphenyls												
9	208	208L	54:33	1.0003	0.9994-1.0012	6	208L	455	1000	46	100	50
9	207	208L	55:32	1.0183	1.0174-1.0193	6	208L/206L	453	1000	45	100	50
9	206	206L	59:37	1.0003	0.9994-1.0011	6	206L	451	1000	45	100	50
Decachlorobiphenyl												
10	209	209L	61:15	1.0003	0.9995-1.0011	6	209L	153	500	15	50	20
Labeled Compounds												
7	188L	194L	41:51	0.7304	0.7275-0.7333	20	194L					
7	180L	194L	50:27	0.8805	0.8775-0.8834	20	194L					
7	170L	194L	51:53	0.9055	0.9026-0.9084	20	194L					
7	189L	194L	55:06	0.9616	0.9587-0.9645	20	194L					
8	202L	194L	47:31	0.8293	0.8264-0.8322	20	194L					
8	205L	194L	57:48	1.0087	1.0044-1.0131	30	194L					

Cl No.[8]	IUPAC No. [9,10]	RT Ref[11]	RTs[12]	RRT[13]	RRT Limits[14]	Window (sec)[15]	Quantitation Reference[16]	Estimated Detection and Quantitation Limits - Matrix and Concentration[17]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
9	208L	194L	54:32	0.9517	0.9488-0.9546	20	194L					
9	206L	194L	59:36	1.0401	1.0358-1.0445	30	194L					
10	209L	194L	61:14	1.0686	1.0643-1.0730	30	194L					
Labeled Cleanup Standards												
3	28L	52L	26:44	0.9266	0.9209-0.9324	20	52L					
5	111L	101L	38:51	1.0777	1.0730-1.0823	20	101L					
7	178L	138L	45:05	1.0090	1.0052-1.0127	20	138L					
Labeled Internal Standards												
2	9L	138L	18:54	0.4648	0.4596-0.4699	25	178L					
4	52L	138L	28:51	0.7094	0.7043-0.7145	25	178L					
5	101L	138L	36:03	0.8865	0.8814-0.8916	25	178L					
6	138L	138L	44:41	1.0988	1.0783-1.1193	100	178L					
8	194L	138L	57:18	1.4090	1.4039-1.4141	25	178L					

Table 3. Concentrations of Native and Labeled Chlorinated Biphenyls in Stock Solutions, Spiking Solutions, and Final Extracts

CB Congener	Solution Concentrations Stock (µg/mL)	Spiking (ng/mL)	Extract (ng/mL)
Native Toxics/LOC[18]			
1	20	1.0	50
3	20	1.0	50
4	20	1.0	50
15	20	1.0	50
19	20	1.0	50
37	20	1.0	50
54	20	1.0	50
77	20	1.0	50
81	20	1.0	50
104	20	1.0	50
105	20	1.0	50
114	20	1.0	50
118	20	1.0	50
123	20	1.0	50
126	20	1.0	50
155	20	1.0	50
156	20	1.0	50
157	20	1.0	50
167	20	1.0	50
169	20	1.0	50
188	20	1.0	50
189	20	1.0	50
202	20	1.0	50
205	20	1.0	50
206	20	1.0	50
208	20	1.0	50
209	20	1.0	50
Native Congener Mix Stock Solutions[19]			
MoCB thru TrCB	2.5		
TeCB thru HpCB	5.0		
OcCB thru DeCB	7.5		
Labeled Toxics/LOC/Window-Defining[20]			
1L	1.0	2.0	100
3L	1.0	2.0	100
4L	1.0	2.0	100
15L	1.0	2.0	100
19L	1.0	2.0	100
37L	1.0	2.0	100
54L	1.0	2.0	100
77L	1.0	2.0	100
81L	1.0	2.0	100
104L	1.0	2.0	100
105L	1.0	2.0	100
114L	1.0	2.0	100
118L	1.0	2.0	100
123L	1.0	2.0	100
126L	1.0	2.0	100
155L	1.0	2.0	100
156L	1.0	2.0	100
157L	1.0	2.0	100
167L	1.0	2.0	100
169L	1.0	2.0	100
188L	1.0	2.0	100
189L	1.0	2.0	100
202L	1.0	2.0	100

CB Congener	Solution Concentrations		
	Stock (µg/mL)	Spiking (ng/mL)	Extract (ng/mL)
Native Toxics/LOC[18]			
205L	1.0	2.0	100
206L	1.0	2.0	100
208L	1.0	2.0	100
209L	1.0	2.0	100
Labeled Cleanup[21]			
28L	1.0	2.0	100
111L	1.0	2.0	100
178L	1.0	2.0	100
Labeled Injection Internal[22]			
9L	5	1000	100
52L	5	1000	100
101L	5	1000	100
138L	5	1000	100
194L	5	1000	100

Diluted Combined 209-Congener[23]		
	Solution Concentration (µg/mL)	
Standard	**Native**	**Labeled**
Native congeners		
MoCB thru TrCB	50	
TeCB thru HpCB	100	
OcCB thru DeCB	150	
Labeled Toxics/LOC/Window-Defining		100
Labeled Cleanup		100
Labeled Injection internal		100

Table 4. Suggested Composition of Individual Native CB Congener Solutions[24]

Solution Identifier				
A2	**B2**	**C2**	**D2**	**E2**
2	7	13	25	1
10	5	17	21	3
9	12	29	69	4
6	18	20	47	15
8	24	46	42	19
14	23	65	64	16
11	28	59	70	37
30	22	40	102	54
27	39	67	97	43
32	53	76	115	44
34	51	80	123	74
26	73	93	134	56
31	48	84	131	77
33	62	101	163	104
36	71	112	180	98
38	68	86		125
35	58	116		110
50	61	109/107		126
45	55	154		155
52	60	147		138
49	94	140		169
75	100	146		188
41	91	141		189
72	121	164		202
57	90	158		205
63	99	182		208
66	108/109	174		206
79	117	173		209
78	111	193		
81	107/108			
96	118			
103	114			
95	150			
88	145			
89	135			
92	149			
113	139			
83	132			
119	165			
87	168			
85	137			
82	160			
120	128			
124	162			
106	157			
122	184			
105	186			
127	187			
152	185			
136	181			
148	192			
151	197			
144	199/201			
143	203			
142				
133				

Solution Identifier				
A2	**B2**	**C2**	**D2**	**E2**
161				
153				
130				
129				
166				
159				
167				
156				
179				
176				
178				
175				
183				
177				
171				
172				
191				
170				
190				
201/200				
204				
200/199				
198				
196				
195				
194				
207				
Totals				
83	**54**	**29**	**15**	**28**

Table 5. Concentration of CB Congeners in Calibration and Calibration Verification Standards

		Solution Concentration (ng/mL)					
CB Congener	**IUPAC[25]**	**CS0.2 (Hi sens)[26]**	**CS1**	**CS2**	**CS3 (CCV)**	**CS4**	**CS5**
Native Toxics/LOC							
2-MoCB	1	0.2	1.0	5.0	50	400	2000
4-MoCB	3	0.2	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.2	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.2	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.2	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.2	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.2	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.2	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.2	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.2	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.2	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.2	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.2	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.2	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.2	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.2	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.2	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.2	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.2	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.2	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.2	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.2	1.0	5.0	50	400	2000
DeCB	209	0.2	1.0	5.0	50	400	2000
Labeled Toxics/LOC/Window-Defining							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6'-TrCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100

		Solution Concentration (ng/mL)					
CB Congener	IUPAC[25]	CS0.2 (Hi sens)[26]	CS1	CS2	CS3 (CCV)	CS4	CS5
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Labeled Cleanup							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100
Labeled Internal							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100

Table 6. Quality Control (QC) Acceptance Criteria for Chlorinated Biphenyls in Calibration Verification, Initial Precision and Recovery (IPR), and Samples[27]

Congener	IUPAC Number[28]	Test Conc (ng/mL)[29]	Calibration Verification[30] (%)	IPR		Labeled Compound Recovery in Samples
				RSD (%)	X (%)	(%)
2-MoCB	1	50	70-130	40	60-140	
4-MoCB	3	50	70-130	40	60-140	
2,2'-DiCB	4	50	70-130	40	60-140	
4,4'-DiCB	15	50	70-130	40	60-140	
2,2'6-TrCB	19	50	70-130	40	60-140	
3,4,4'-TrCB	37	50	70-130	40	60-140	
2,2'6,6'TeCB	54	50	70-130	40	60-140	
3,3',4,4'-TeCB	77	50	70-130	40	60-140	
3,4,4',5-TeCB	81	50	70-130	40	60-140	
2,2',4,6,6'-PeCB	104	50	70-130	40	60-140	
2,3,3',4,4'-PeCB	105	50	70-130	40	60-140	
2,3,4,4',5-PeCB	114	50	70-130	40	60-140	
2,3',4,4',5-PeCB	118	50	70-130	40	60-140	
2',3,4,4',5-PeCB	123	50	70-130	40	60-140	
3,3',4,4',5-PeCB	126	50	70-130	40	60-140	
2,2',4,4',6,6'-HxCB	155	50	70-130	40	60-140	
2,3,3',4,4',5-HxCB[5]	156	50	70-130	40	60-140	
2,3,3',4,4',5'-HxCB[5]	157	50	70-130	40	60-140	
2,3',4,4',5,5'-HxCB	167	50	70-130	40	60-140	
3,3',4,4',5,5'-HxCB	169	50	70-130	40	60-140	
2,2',3,4',5,6,6'-HpCB	188	50	70-130	40	60-140	
2,3,3',4,4',5,5'-HpCB	189	50	70-130	40	60-140	
2,2',3,3',5,5',6,6'-OcCB	202	50	70-130	40	60-140	
2,3,3',4,4',5,5',6-OcCB	205	50	70-130	40	60-140	
2,2',3,3',4,4',5,5',6-NoCB	206	50	70-130	40	60-140	
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	70-130	40	60-140	
DeCB	209	50	70-130	40	60-140	
$^{13}C_{12}$-2-MoCB	1L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-4-MoCB	3L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2'-DiCB	4L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-4,4'-DiCB	15L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',6-TrCB	19L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	50-150	50	35-135	25-150
13C12-3,3',4,4'-TCB	77L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	50-150	50	35-135	25-150

Congener	IUPAC Number[28]	Test Conc (ng/mL)[29]	Calibration Verification[30] (%)	IPR		Labeled Compound Recovery in Samples
				RSD (%)	X (%)	(%)
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3,3',4,4',5 -HxCB[31]	156L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[31]	157L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB	189L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50-150	50	35-135	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50-150	50	35-135	25-150
Cleanup Standard						
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	60-130	45	45-120	30-135
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	60-130	45	45-120	30-135
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	60-130	45	45-120	30-135

Table 7. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z	m/z Type	m/z Formula	Substance
Fn-1; Cl-1	188.0393	M	$^{12}C_{12}$ H_9 ^{35}Cl	Cl-1 CB
	190.0363	M+2	$^{12}C_{12}$ H_9 ^{37}Cl	Cl-1 CB
	200.0795	M	$^{13}C_{12}$ H_9 ^{35}Cl	$^{13}C_{12}$ Cl-1 CB
	202.0766	M+2	$^{13}C_{12}$ H_9 ^{37}Cl	$^{13}C_{12}$ Cl-1 CB
	218.9856	lock	C_4 F_9	PFK
Fn-2; Cl-2,3	222.0003	M	$^{12}C_{12}$ H_8 $^{35}Cl_2$	Cl-2 PCB
	223.9974	M+2	$^{12}C_{12}$ H_8 ^{35}Cl ^{37} Cl	Cl-2 PCB
	225.9944	M+4	$^{12}C_{12}$ H_8 $^{37}Cl_2$	Cl-2 PCB
	234.0406	M	$^{13}C_{12}$ H_8 $^{35}Cl_2$	$^{13}C_{12}$ Cl-2 PCB
	236.0376	M+2	$^{13}C_{12}$ H_8 ^{35}Cl ^{37} Cl	$^{13}C_{12}$ Cl-2 PCB
	242.9856	lock	C_6 F_9	PFK
	255.9613	M	$^{12}C_{12}$ H_7 $^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}$ H_7 $^{35}Cl_2$ ^{37}Cl	Cl-3 PCB
Fn-3	255.9613	M	$^{12}C_{12}$ H_7 $^{35}Cl_3$	Cl-3 PCB
Cl-3,4,5	257.9584	M+2	$^{12}C_{12}$ H_7 $^{35}Cl_2$ ^{37}Cl	Cl-3 PCB
	259.9554	M+4	$^{12}C_{12}$ H_7 ^{35}Cl $^{37}Cl_2$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}$ H_7 $^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}$ H_7 $^{35}Cl_2$ ^{37}Cl	$^{13}C_{12}$ Cl-3 PCB
	280.9825	lock	C_6 F_{11}	PFK
	289.9224	M	$^{12}C_{12}$ H_6 $^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}$ H_6 $^{35}Cl_3$ ^{37}Cl	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}$ H_6 $^{35}Cl_2$ $^{37}Cl_2$	Cl-4 PCB
	301.9626	M	$^{13}C_{12}$ H_6 $^{35}Cl_4$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{13}C_{12}$ H_6 $^{35}Cl_3$ ^{37}Cl	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}$ H5 $^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}$ H5 $^{35}Cl_4$ ^{37}Cl	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}$ H5 $^{35}Cl_3$ $^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}$ H5 $^{35}Cl_4$ ^{37}Cl	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}$ H5 $^{35}Cl_3$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
Fn-4	289.9224	M	$^{12}C_{12}$ H_6 $^{35}Cl_4$	Cl-4 PCB
Cl-4,5,6	291.9194	M+2	$^{12}C_{12}$ H_6 $^{35}Cl_3$ ^{37}Cl	Cl-4 PCB

Function and Chlorine Level	m/z	m/z Type	m/z Formula	Substance
	293.9165	M+4	$^{12}C_{12}$ H_6 $^{35}Cl_2$ $^{37}Cl_2$	Cl-4 PCB
	301.9626	M+2	$^{13}C_{12}$ H_6 $^{35}Cl_3$ ^{37}Cl	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+4	$^{13}C_{12}$ H_6 $^{35}Cl_2$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}$ H_5 $^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}$ H_5 $^{35}Cl_4$ ^{37}Cl	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}$ H_5 $^{35}Cl_3$ $^{37}Cl_2$	Cl-5 PCB
	330.9792	lock	C_7 F_{15}	PFK
	337.9207	M+2	$^{13}C_{12}$ H_5 $^{35}Cl_4$ ^{37}Cl	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}$ H_5 $^{35}Cl_3$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	359.8415	M+2	$^{13}C_{12}$ H_4 $^{35}Cl_5$ ^{37}Cl	Cl-6 PCB
	361.8385	M+4	$^{13}C_{12}$ H_4 $^{35}Cl_4$ $^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{13}C_{12}$ H_4 $^{35}Cl_3$ $^{37}Cl_2$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}$ H_4 $^{35}Cl_5$ ^{37}Cl	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}$ H_4 $^{35}Cl_4$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
Fn-5	323.8834	M	$^{12}C_{12}$ H_5 $^{35}Cl_5$	Cl-5 PCB
Cl-5,6,7	325.8804	M+2	$^{12}C_{12}$ H_5 $^{35}Cl_4$ ^{37}Cl	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}$ H_5 $^{35}Cl_3$ $^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}$ H_5 $^{35}Cl_4$ ^{37}Cl	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}$ H_5 $^{35}Cl_3$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	354.9792	lock	C_9 F_{13}	PFK
	359.8415	M+2	$^{12}C_{12}$ H_4 $^{35}Cl_5$ ^{37}Cl	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}$ H_4 $^{35}Cl_4$ $^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}$ H_4 $^{35}Cl_3$ $^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}$ H_4 $^{35}Cl_5$ ^{37}Cl	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}$ H_4 $^{35}Cl_4$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
	393.8025	M+2	$^{12}C_{12}$ H_3 $^{35}Cl_6$ ^{37}Cl	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}$ H_3 $^{35}Cl_5$ $^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}$ H_3 $^{35}Cl_4$ $^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}$ H_3 $^{35}Cl_6$ ^{37}Cl	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}$ H_3 $^{35}Cl_5$ $^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	454.9728	QC	C_{11} F_{17}	PFK
Fn-6	393.8025	M+2	$^{12}C_{12}$ H_3 $^{35}Cl_6$ ^{37}Cl	Cl-7 PCB

Function and Chlorine Level	m/z	m/z Type	m/z Formula	Substance
Cl-7,8,9,10	395.7995	M+4	$^{12}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}\ H_3\ ^{35}Cl_4\ ^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}\ H_3\ ^{35}Cl_6\ ^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	427.7635	M+2	$^{12}C_{12}\ H_2\ ^{35}Cl_7\ ^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}\ H_2\ ^{35}Cl_6\ ^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}\ H_2\ ^{35}Cl_5\ ^{37}Cl_3$	Cl-8 PCB
	439.8038	M+2	$^{13}C_{12}\ H_2\ ^{35}Cl_7\ ^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}\ H_2\ ^{35}Cl_6\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	442.9728	QC	$C_{10}\ F_{13}$	PFK
	454.9728	lock	$C_{11}\ F_{13}$	PFK
	461.7246	M+2	$^{12}C_{12}\ H_1\ ^{35}Cl_8\ ^{37}Cl$	Cl-9 PCB
	463.7216	M+4	$^{12}C_{12}\ H_1\ ^{35}Cl_7\ ^{37}Cl_2$	Cl-9 PCB
	465.7187	M+6	$^{12}C_{12}\ H_1\ ^{35}Cl_6\ ^{37}Cl_3$	Cl-9 PCB
	473.7648	M+2	$^{13}C_{12}\ H_1\ ^{35}Cl_8\ ^{37}Cl$	$^{13}C_{12}$ Cl-9 PCB
	475.7619	M+4	$^{13}C_{12}\ H_1\ ^{35}Cl_7\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-9 PCB
	495.6856	M+2	$^{13}C_{12}\ H_4\ ^{35}Cl_9\ ^{37}Cl$	Cl-10 PCB
	497.6826	M+4	$^{12}C_{12}\ ^{35}Cl_8\ ^{37}Cl_2$	Cl-10 PCB
	499.6797	M+6	$^{12}C_{12}\ ^{35}Cl_7\ ^{37}Cl_3$	Cl-10 PCB
	507.7258	M+2	$^{13}C_{12}\ H_4\ ^{35}Cl_9\ ^{37}Cl$	$^{13}C_{12}$ Cl-10 PCB
	509.7229	M+4	$^{13}C_{12}\ H_4\ ^{35}Cl_8\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-10 PCB
	511.7199	M+6	$^{13}C_{12}\ H_4\ ^{35}Cl_8\ ^{37}Cl_4$	$^{13}C_{12}$ Cl-10 PCB

1. Isotopic masses used for accurate mass calculation

^{1}H	1.0078
^{12}C	12.0000
^{13}C	13.0034
^{35}Cl	34.9689
^{37}Cl	36.9659
^{19}F	18.9984

Exhibit D CB Congeners -- Section 17
Tables/Diagrams/Flowcharts (Con’t)

Table 8. Theoretical Ion Abundance Ratios and QC Limits

Chlorine Atoms	**m/z's Forming Ratio**	**Theoretical Ratio**	**Lower QC Limit**	**Upper QC Limit**
1	m/m+2	3.13	2.66	3.60
2	m/(m+2)	1.56	1.33	1.79
3	m/(m+2)	1.04	0.88	1.20
4	m/(m+2)	0.77	0.65	0.89
5	(m+2)/(m+4)	1.55	1.32	1.78
6	(m+2)/(m+4)	1.24	1.05	1.43
7	(m+2)/(m+4)	1.05	0.89	1.21
8	(m+2)/(m+4)	0.89	0.76	1.02
9	(m+2)/(m+4)	0.77	0.65	0.89
10	(m+2)/(m+4)	0.69	0.59	0.79

Endnotes:

1. Abbreviations for chlorination levels:
   MoCB = monochlorobiphenyl
   DiCB = dichlorobiphenyl
   TrCB = trichlorobiphenyl
   TeCB = tetrachlorbiphenyl
   PeCB = pentachlorobiphenyl
   HxCB = hexachlorobiphenyl
   HpCB = heptachlorobiphenyl
   OcCB = octachlorobiphenyl
   NoCB = nonachlorobiphenyl
   DeCB = decachlorobiphenyl
2. Labeled Level of Chlorination (LOC) Window-Defining Congener.
3. National Oceanic and Atmospheric Administration (NOAA) Congener of Interest.
4. Labeled Internal Standard.
5. Labeled Cleanup Standard.
6. World Health Organization (WHO) Toxic Congener.
7. Labeled analog of WHO Toxic Congener.
8. Number of chlorines on congener.
9. Suffix "L" indicates labeled compound.
10. Multiple congeners in a box indicates a group of congeners that coelute or may not be adequately resolved on a 30-m SPB-Octyl column. Congeners included in the group are listed as the last entry in the box.
11. Retention Time (RT) reference that is used to locate target congener.
12. RT of target congener.
13. Relative Retention Time (RRT) between the RT for the congener and RT for the reference.
14. Nominal limits based on an ± 0.5% of the RRT, adjusted for the nearest eluted isomer.
15. RT window width for congener or group of two or more congeners.
16. Labeled congeners that form the quantitation reference. Areas from the exact m/z ratios of the congeners listed in the quantitation reference are summed, and divided by the number of congeners in the quantitation reference. For example, for CB 10, the areas at the exact m/z ratios for 4L and 15L are summed and the sum is divided by 2 (because there are two congeners in the quantitation reference).
17. Detection Limits and Contract Required Quantitation Limits (CRQLs) with common laboratory interferences present. Without interferences, EMDLs and EMLs will be, respectively, 5 and 10 pg/L for aqueous samples, 0.5 and 1.0 ng/kg for soil, tissue, and mixed-phase samples, and EMLs for extracts will be 0.5 pg/uL.
18. Stock solution: Section 7.8.1; Spiking solution: Section 7.11.
19. Section 7.8.2.
20. Stock solution: Section 7.9.1; Spiking solution: Section 7.12.
21. Stock solution: Section 7.9.2; Spiking solution: Section 7.13.
22. Stock solution: Section 7.9.3; Spiking solution: Section 7.14.
23. Section 7.10.2.2.
24. Congeners present in each standard listed in elution order for each level of chlorination. IUPAC Number listed first; Ballschmiter (BZ) Number listed second where ambiguous. See Table 3 for concentrations of congeners in stock solutions and Table 5 for concentrations in Calibration Standard.
25. Suffix "L" indicates labeled compound.
26. Additional concentration used for calibration of high sensitivity HRGC/HRMS systems.
27. QC acceptance criteria for IPR, and samples based on a 20 µL extract final volume.
28. Suffix "L" indicates labeled compound.
29. See Table 5.
30. Section 9.
31. PCBs 156 and 157 are tested as the sum of two concentrations.